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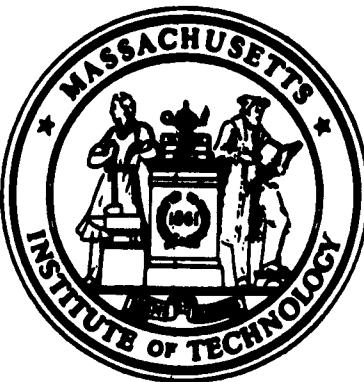
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SOIL STABILIZATION

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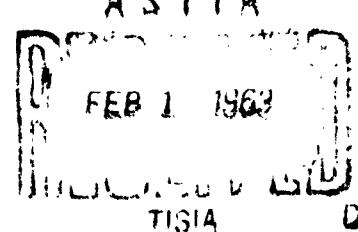


MASSACHUSETTS INSTITUTE of TECHNOLOGY

For

CORPS OF ENGINEERS, WATERWAYS EXPERIMENT STATION

FINAL REPORT, PHASE XIII
NOVEMBER, 1961



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by
CHEMICAL METHODS**

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INTRODUCTION

A. Contract

Since 18 June 1946, the Massachusetts Institute of Technology Soil Stabilization Laboratory has been conducting research on soil stabilization under the sponsorship of the Corps of Engineers, U.S. Army. The work has been covered by the following contracts:

W-44009-ENG-408	18 June 1948 - 15 March 1950
DA-44-009-ENG-11	15 March 1950 - 15 March 1951
DA-44-009-ENG-924	15 October 1951 - 15 October 1952
DA-44-009-ENG-1494	21 October 1952 - 22 October 1953
DA-44-009-ENG-2002	22 October 1953 - 22 October 1954
DA-22-079-ENG-171	22 October 1954 - 22 October 1960
DA-22-079-ENG-288	22 October 1960 - 21 October 1961

This report entitled, "Soil Stabilization by Chemical Methods," Final Report No. XIII describes work done under Contract No. DA-22-079-ENG-288 during the period 22 October 1960 to 21 October 1961.

B. Scope of Contract

Article 1 of Contract No. DA-22-079-ENG-288 states the work and services to be performed. Below are listed those parts of this report which describe work on contract items:

Item No. in Article 1	Report Section
(b)	II, III
(c)	VI
(d)	II, III, IV, V
(e)	VII
(f)	II, III, IV, V, VII, VIII

In addition to funds from Contract No. DA-22-079-ENG-288, the Soil Stabilization Laboratory is sponsored by funds from industrial organizations. These funds support fundamental and applied soil stabilization research unrestricted in scope. When work supported by those non-Government funds is thought to be of value to the Army-sponsored work, it is included in the reports made to the Army.

C. Organization

The soil stabilization research was conducted by the Massachusetts Institute of Technology Soil Stabilization Laboratory. The laboratory, which is a co-operative venture between the Departments of Civil Engineering and Chemical Engineering at M.I.T., has the following organization and staff:

Director	Dr. T. William Lambe, Professor and Head, Soil Engineering Division, Department of Civil Engineering
Associate Director	Dr. Alan S. Michaels, Professor of Chemical Engineering

The staff of the Laboratory consisted of many part-time workers and the following:

Dr. R. T. Martin	Research Associate, Dept. of Civil Engineering
Dr. C. C. Ladd	Assistant Professor, Dept. of Civil Engineering
Mr. F. W. Tausch	Research Assistant, Dept. of Chemical Engineering (Started September, 1956)
Mr. R. F. Friesecke	Research Assistant, Dept. of Civil Engineering (Started September, 1960)
Mr. A. S. Kizraglon	Research Assistant, Dept. of Chemical Engineering (Started September, 1960)
Mr. R. P. Merrill	Research Assistant, Dept. of Chemical Engineering (Started September, 1960)
Mr. N. J. Pannu	Research Assistant, Dept. of Chemical Engineering (Started September, 1960)
Mr. A. E. Z. Wissa	Research Assistant, Dept. of Civil Engineering (Started September, 1960)

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I. SUMMARY

A. Soil Stabilization with Acidic Phosphorous Compounds and Secondary Additives

Studies of stabilization of two fine grained soils with mixtures of phosphate rock and sulfuric acid (phosphate equivalent to 2% H_3PO_4) have been continued, with emphasis on reducing stabilization costs and increasing strength and water resistance. The soils studied were the same as those used in previous work, i.e., Massachusetts Clayey Silt (MCS) and Vicksburg Buckshot Clay (VBC). Major variables studied included 1) total fluids content at compaction, 2) "free" sulfuric acid concentration, i.e., in excess of that required for stoichiometric reaction with the phosphate rock, 3) octylamine (waterproofer) concentration, and 4) sodium fluosilicate (cure accelerator) concentration.

The results with both soils, and with all additive formulations, indicate that humid cure strength increases with liquids content up to an optimum liquids content (about 12 cc liquids/100 gms soil for MCS, and about 19-20 cc/100 gms for VBC), above which humid cure strength drops off with increasing liquids content. Immersed strengths, however, increase with liquids content up to the highest levels which could be achieved, apparently due to more complete soil saturation before immersion and hence less tendency to absorb water.

Limited concentrations of "free" sulfuric acid (up to about 1.4% H_2SO_4 in excess of the 2.6% required for stoichiometric reaction) were found to significantly increase both humid cure strength and immersed strength of both soils. For example, with all other conditions approximately constant, addition of 1.4% "free" sulfuric acid increased one day humid cure strength of MCS from 312 to 425 psi, and increased seven day immersed strength from 80 to 270 psi. Higher concentrations of "free" sulfuric acid (i.e., 2.4%) were found to give very low strengths for all cure conditions. These effects are consistent with the proposed mechanism of stabilization; limited concentrations of "free" sulfuric acid apparently increase the rate of dissolution of alumina and hence provide additional cementitious material, while high concentrations of "free" acid prevent gelation of the cementitious material.

Limited work indicated that sulfuric acid alone (without phosphate rock) also has a stabilizing effect, presumably due to cementation by colloidal alumina. The beneficial effects of "free" sulfuric acid may therefore be due, at least in part, to this additional stabilization.

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The waterproofing effect of octylamine was further confirmed, although with 1.4% "free" sulfuric acid and liquids content near or above optimum satisfactory seven day immersed strengths (i.e., 270 psi) can be achieved with MCS without octylamine. For satisfactory one day immersed strengths, however, 0.5% octylamine was required even with MCS. The results also suggest that Armeen D at a concentration of 0.05% may be as effective as 0.5% octylamine, although this is not certain as higher fluids contents were used with the Armeen D. Octylamine (or Armeen D) was used at 1% concentration in all the work with VBC.

The results indicate that sodium fluosilicate does not accelerate the cure of MCS samples stabilized with mixtures of phosphate rock and sulfuric acid, presumably because of the fact that the phosphate rock itself contains considerable fluoride. Fluosilicate, at 0.5%, was found to accelerate cure of VBC somewhat, however.

Other significant observations included the following: 1) two different batches of phosphate rock are equally effective; 2) octylamine, when used, must be dissolved in diluted sulfuric acid to prevent degradation; and 3) phosphate rock and sulfuric acid must be preblended several minutes before addition to the soil, to permit escape of evolved gases before compaction of samples.

B. Stabilization of Soils with Lime

Research during this period was aimed at clarifying further the soil-lime-additive stabilization mechanism, investigating the effectiveness of several inorganic compounds as secondary additives, and evaluating CaO as a stabilizer for high plasticity soils.

Three high plasticity soils were used in this investigation, including VBC on which the major part of the work was done. The results obtained with these soils indicated that CaO stabilization depends on the reactive silica contained in the montmorillonoid and is hence related to the amount of montmorillonoid mineral in the soil.

Of the additives used, $MgSO_4$, $ZnSO_4$, and $NiSO_4$ produced approximately equal improvements in strength when used alone. When used in conjunction with Na_2SiO_3 , however, $MgSO_4$ produced the greatest improvement in strength.

Measurements of the pH of aqueous solutions of CaO, and CaO plus $MgSO_4$ at different concentrations, have shown that $MgSO_4$ has

no effect on pH. This, and the previous observations with metasilicate and the other inorganic salts, support the view that the improvement in strength of CaO-stabilized soils caused by the above mentioned additives is not due to additional solubilization of soil silica, but rather to the formation of cementitious gels with properties superior to calcium silicate.

C. Soil Stabilization with Asphalt Emulsions

The present work in asphalt stabilization has been directed toward development of load-bearing strength, water resistance, and water-impermeability of sandy soils (e.g., Ottawa Sand) by topical treatment with asphalt emulsions. The effect of various application variables has been studied.

All emulsions prepared with gasoline as a cutback solvent produced deleterious soil foams, but an emulsion prepared with a solvent containing no light ends (50-50 mixture n-heptane and toluene) did not produce these foams and the study of application techniques was continued with this emulsion.

To date, the effects of dilution of the emulsion and of prewetting the soil with water have been studied. Regardless of the application technique, however, fully-cured penetration strengths of 50 psi were obtained. Depths of asphalt penetration varied from 12 to 25 mm for a dosage of 0.135 gms asphalt/cm² soil, and were greatest for pre-wet soils and diluted emulsions. Curing times were greatest for undiluted emulsions applied to dry soils, and shortest for pre-wet soils. Samples prepared with diluted emulsions and those prepared by pre-wetting, however, had permeabilities from 10 to 100 times as great as samples prepared by applying undiluted emulsions to dry soil.

D. Soil Stabilization with an Iron-Aluminum Salt of Phosphoric Acid (TCB) (abstracted from an S. B. Thesis by H. H. Bawany)

The objective of this work was to investigate the effectiveness of a potential soil stabilizer known as TCB on two soils, Massachusetts Clayey Silt and Vicksburg Buckshot Clay. Since TCB contains phosphorous pentoxide (stabilizing agent), iron and aluminum oxides (waterproofing agents), and fluorine (cure accelerator), it was expected that successful stabilization would result.

The experimental procedure involved dry-mixing of the soils with TCB, blending of the mixture with additional water in a mechanical mixer, compaction, curing, and testing of samples.

It was found that Massachusetts Clayey Silt could be successfully stabilized with TCB. At 6% TCB on dry soil weight, equivalent to 2.4% phosphoric acid, the compressive strengths obtained were comparable to those obtained with 2% phosphoric acid alone. The fluorine in TCB had a significant effect on cure acceleration.

Vicksburg Buckshot Clay could not be successfully stabilized with TCB alone. Iron and aluminum oxides in TCB had no significant effect on this soil.

Economic considerations indicated that TCB is not at present competitive with other stabilizing systems for Massachusetts Clayey Silt, and entirely unsuitable for Vicksburg Buckshot Clay.

E. Evaluation of Soil Stabilizers

One objective of this work was to ascertain the most economical stabilizers that have been developed for three soils (Massachusetts Clayey Silt, M-21; Vicksburg Buckshot Clay, V. B. C.; and New Hampshire Silt, N. H. S.) based on the data contained in past M. I. T. Soil Stabilization reports. Strength criteria selected for the comparison were unconfined compressive strengths of 150 and 300 psi after 7 days humid cure and one day immersion. These strength values were chosen as possible minimum values for subbase and base course stabilization for secondary roads. This review of existing data showed the following systems to be the most economical:

<u>Soil</u>	<u>For 150 psi</u>		<u>For 300 psi</u>	
	<u>System</u>	<u>Cost(\$/ yd³)</u>	<u>System</u>	<u>Cost(\$/ yd³)</u>
V. B. C.	2% Quicklime	0.40	4.6% Quicklime) 1.2% MgSO ₄)	1.57
M-21	2.4% Portland) Cement)	1.07	5.0% Portland) Cement)	2.20
N. H. S.	5.0% Portland) Cement) 0.2% Na ₂ SO ₄)	1.95	5.0% Portland) Cement) 1.2% Na ₂ SO ₄)	2.60

It should be pointed out, however, that the data available did not always allow determination of the most economical formulations. Moreover, the selection of the strength values was quite arbitrary. The values

should have been varied according to soil type and type of stabilization. For example, an asphalt stabilized soil would not require as high an unconfined strength as a cement or lime stabilized soil for satisfactory performance.

The second objective was to investigate the effects of compactive effort, type of compaction, time of humid cure, cycles of wetting and drying, and cycles of freezing and thawing on the soaked unconfined compressive strength of soils stabilized with economical formulations yielding strength values of 150 and 300 psi. Four soil-additive systems were chosen:

1. M-21 plus 5% portland cement (300 psi)
2. M-21 plus 1.5% phosphoric acid (150 psi)
3. VBC plus 5% quicklime and 1.2% magnesium sulfate (300 psi)
4. VBC plus 4% quicklime (150 psi)

The results of the cyclic tests were most interesting and suggest the following for the soil-additive systems evaluated:

1. A strength of 300 psi for base course construction is adequate under all conditions with stabilized M-21 but definitely too low with treated VBC if subjected to wetting and drying and somewhat marginal if this clay is subjected to freezing and thawing.
2. A strength of 150 psi for subbase construction is definitely too low for stabilized VBC if subjected to wetting and drying and is marginal for both VBC and M-21 if cycles of freezing and thawing occur. However, stabilized M-21 with a strength of 150 psi did withstand cycles of wetting and drying.

It was concluded that the values of soaked unconfined compressive strength selected as design criteria for "cemented" stabilized soils should be varied according to the type of soil and the type of climate.

F. Effective Stresses in Stabilized Soils

The effective stress principle has been neglected until now in chemical stabilization work in favor of easier, quicker methods which, although sometimes reliable, have not provided the tools so necessary for a fundamental analysis of what the stabilizers actually do to the soil. By understanding how stabilizers interact with soil to generate greater strength one is in a better position to evaluate the effectiveness

of a stabilizer and to develop more effective and more versatile stabilizers. The work described in this section represents the initial phases of this new investigation into the strength behavior of compacted and of stabilized soils in terms of effective stresses. One phase concerns effective stresses and strength in a partially saturated unstabilized compacted clay and the other presents data on stress-strain curves and effective stress Mohr envelopes for a saturated, stabilized clay.

Equipment and testing procedures are described for measuring residual pore water pressures in compacted samples utilizing a fine ceramic stone and a pressure transducer for measuring pore water pressures. Values of residual pore water pressure over a wide molded water content range for a quartz-kaolinite mixture are reported along with data showing the relationship between pore air pressure, "capillary" pressure, and pore water pressure.

Strength data are reported from consolidated-undrained triaxial tests with pore pressure measurements on saturated samples of a compacted quartz-kaolinite mixture. The molded water content was varied so as to yield different values of residual pore water pressure. Each specimen was subsequently saturated with an effective consolidation pressure equal to the value of residual pore water pressure after molding. Of interest, all specimens yielded the same Mohr envelopes in terms of effective stresses although the undrained strengths varied considerably.

A fat clay (Vicksburg Buckshot Clay) treated with 5% hydrated lime was selected for study with consolidated-undrained triaxial tests with pore pressure measurements. After curing and immersion the specimens were saturated with consolidation pressures ranging from zero to 6 kg/cm^2 . The tests showed that, relative to normally consolidated clay, lime increased the cohesion intercept of the Mohr envelope in terms of effective stresses from 0 to 1.6 kg/cm^2 and the friction angle from 23° to 32.5° . Moreover, none of the samples developed large negative pore pressures during shear.

II. SOIL STABILIZATION WITH ACIDIC PHOSPHORUS COMPOUNDS AND SECONDARY ADDITIVES

A. Introduction

Research during the past several years has shown that phosphoric acid and other acidic phosphorus compounds, in combination with various secondary additives, are effective for stabilization of a variety of fine-grained soils. Among the major developments have been: 1) the use of fluorine compounds (e.g., sodium fluosilicate) to accelerate cure; 2) the use of amines (e.g., octylamine) to improve water resistance; 3) the use of mixtures of inexpensive phosphate rock and sulfuric acid to generate phosphoric acid in the soil and thereby greatly reduce stabilization costs; and 4) development of postulated mechanisms of stabilization which are consistent with experimental results (references 1-4).

The objective of the research carried out during the period covered by this report (October 1960 - October 1961) has been to further reduce stabilization costs and improve the strength and water resistance of two widely different soils (Massachusetts Clayey Silt, a lean soil with a plasticity index of 6, and Vicksburg Buckshot Clay, a fatty soil with a plasticity index of about 40) with acidic phosphorus compounds and secondary additives.

Specific objectives included: 1) study of the effect of total fluid content at mixing on strength; 2) study of the effect of excess sulfuric acid on strength development; 3) study of the effect of octylamine on water resistance; and 4) study of the effect of sodium fluosilicate on rate of cure.

B. Experimental

1. Materials

The physical and chemical properties of the soils used in this investigation are presented in Table II-1. The Massachusetts Clayey Silt (MCS) was a different batch from that used in previous work, but was obtained from the same location (within a few feet) and was found to have very similar properties. The Vicksburg Buckshot Clay (VBC) was obtained from a location several miles from the source of previous batches (Reference 5).

The phosphoric acid used was Merck, reagent grade, analyzing 86% H_3PO_4 . The sulfuric acid was DuPont, reagent grade (98% H_2SO_4 , specific gravity 1.84). The sodium fluosilicate was also reagent grade.

Octylamine from two sources was used. Normal-octylamine (Pennsalt Chemicals), the same batch as that used in previous work, was used in most cases. Armour's Armeen 8 (technical grade n-octylamine) was used in some cases where noted.

Different batches of phosphate rock were also used. Both were supplied by International Minerals and Chemicals Co., were in the form of fine powders, and had comparable phosphate content, some carbonates, and about 3% combined fluorine. The specific properties of the two batches are presented in Table II-2. Batch I was the same as that used in previous work, and was used in most of the present work.

2. Preparation and Testing of Samples

The preparation and testing of samples were similar to previous work. MCS was equilibrated with about 6% water and VBC was equilibrated at about 11% water. Chemicals and additional water were then slowly added and distributed while mixing was carried out in a finger-prong mixer, and mixing was continued for 3 to 4 minutes.

Sodium fluosilicate, when used, was always mixed with the soil before addition of the primary additives.

When phosphate rock-sulfuric acid mixtures were used, the rock and sulfuric acid were pre-blended and the slurry added to the soil while mixing. In most cases (unless otherwise noted) the sulfuric acid was diluted with the additional water before slurring the rock.

When octylamine was used, it was reacted with the sulfuric acid to form a clear solution, then blended with the phosphate rock before adding to the soil. In most cases (unless otherwise noted) the sulfuric acid was diluted with an approximately equal volume of water prior to adding the octylamine; when a large amount of octylamine was added to undiluted sulfuric acid, the solution became very hot and black, indicating degradation of the octylamine. The few cases where this degradation occurred are noted in the tabulated results and discussion.

After mixing, samples were prepared by two-end static compaction at 1000 psi (unless otherwise noted) in a Harvard miniature mold.

Samples were cured at 100% relative humidity for various periods of time (1, 7, 28 days), then either tested (humid cure strength) or immersed in water for one day prior to testing (immersed strength). For example, 7-days immersed strength involved 7 days humid cure followed by one day immersion.

Samples were tested in unconfined compression at a constant, slow rate of strain.

Samples were weighed and measured at the time of compaction and at the time of testing. After testing, samples were dried for 24 hours at 110°C and then weighed.

From the measurements, the following were calculated:

- 1.) Total fluid content at molding (measured), cc/ 100 grams dry soil, as determined from the initial soil water content and the volume of the fluids added.
- 2.) Volatile content at molding, gms/ 100 gms. dry solids, as determined from the weight loss on drying and the weight at molding.

3.) Volatile content at testing, gms/ 100 gms dry solids, as determined from the weight loss on drying and the weight at testing.

4.) Dry density at test, pounds solids / ft³, as determined from the volume at test and the dry weight.

5.) Compressive strength, psi, as determined from the load at failure and the area of the sample at test.

6.) Fluids content at molding (calculated), cc/ 100 gms dry solids, as determined from the volatile content at molding and the volume of fluids added (assuming that one mole of water is released when a mole of sulfuric acid reacts with an equivalent amount of phosphate).

C. Results and Discussion

1. Effect of Fluids Content on Strength and Water Resistance

As previously mentioned, fluids content at molding was determined in two ways. First, the measured fluid content at molding was determined by the initial water content of the soil and the volume of fluids added. Second, as a check, the calculated fluid content at molding was determined from the volatile content of the samples at time of test and

the volume of sulfuric acid added (which is non-volatile), assuming that one mole of water is released when a mole of sulfuric acid reacts with an equivalent amount of phosphoric acid. Because the sulfuric acid is non-volatile at 110°C the fluids content at molding would be expected to be higher than the volatile content at molding (i.e., by 0.93 gm/100 gms soil for no free sulfuric acid, by 1.44 for 1.4% free acid, by 1.79 for 2.4% free acid, and by 2.2 for 3.4% free acid).

One would expect reasonably good agreement between the measured and the calculated fluids content at molding. The results in Tables II-3 and II-4, however, show that the calculated fluids content is considerably less than the measured fluids content; this discrepancy ranges up to 3 cc/100 gms dry soil for MCS, and up to about 5 cc/100 gms dry soil for VBC. The magnitude of this discrepancy appears to be random (i.e., does not correlate with acid concentration), and it is difficult to account for it completely although a number of factors could contribute to an apparent loss of fluids content. First, the over-all reaction of sulfuric acid with phosphate rock and alumina in the soil to form aluminophosphate gel and $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ results in a net loss of water which can account for about 0.2 cc fluids content per 100 gms soil tied up in the calcium sulfate and not volatile below 128°C. A few samples were further dried at 150°C for 24 hours, and the fact that these samples lost several additional per cent in weight is consistent with the postulate of chemically bound water except that the loss on further drying is greater than 0.2%. A second possible explanation is that unreacted sulfuric or phosphoric acid in the soil retains some weight on drying at 110°C, because of the depression of water vapor pressure. A third possibility is that the samples may not have been completely dried by 24 hours at 110°C, which would result in low values for the volatile content and hence low calculated fluids content; the fact that several samples which were dried for an additional 24 hours lost no additional weight seems to indicate that drying is complete in 24 hours. A fourth possibility, and perhaps most important, is that a significant amount of water may be lost by evaporation during mixing and molding of samples. It is quite conceivable that a loss of one or two per cent of water content occurred in this way, although losses of 5 per cent with VBC are more difficult to justify. It is interesting to note, however, that the MCS losses amount to about 3/12 or 25% of the total fluids content, while the VBC losses amount to about 5/20 or also about 25%, which lends some support to the explanation involving evaporation of water during mixing and molding.

In any event, it should be noted that the measured fluids content at molding would be expected to be more meaningful than the value calculated from the measured volatile content, and the interpretation

of results will therefore be based primarily on the measured fluids content values.

Previous work has shown that humid cure strength and compacted density generally increase with increasing fluids content, due to the lubricating effect of increased fluids and other factors. Above a certain fluids content, however, the soil approaches saturation and additional fluids reduce density and strength.

The results in Table II-3 show that, for a given stabilization formulation, humid cure strength and density of MCS tend to increase with fluids content at low levels. Maximum strength with MCS seems to be obtained at a total fluids content level of about 12.5 cc/ 100 gms dry soil, which is consistent with previous work (Reference 2). The results in Tables II-4 and II-5 show the same trends for VBC, with maximum humid cure strength obtained at calculated fluids content of about 19-20 cc/ 100 gms corresponding to a measured fluids content of about 23-24 cc/ 100 gms dry soil.

The results show that immersed strength, on the other hand, generally increases with increasing fluids content up to the maximum fluids content at which samples could be compacted. It may be mentioned here that samples could not be compacted at fluids contents above about 15 cc/ 100 gms dry soil for MCS, or above about 25 cc/ 100 gms dry soil for VBC; above these levels, samples expanded considerably and broke upon removal from the mold, apparently due to air trapped in the samples at these very high fluids content levels. Attempts to avoid entrapment of air by very slow compaction (over a period of several minutes) were promising but not completely successful.

The observed effects of fluids content on strength can be explained as follows. Fluids in the soil aid in compaction (through lubrication) and in distribution of cementitious material. It might be expected, therefore, that compacted density and strength would increase monotonically with increasing fluids content. Above a certain optimum fluids content, however, the soil will approach saturation and additional fluids will interfere with compaction to a high density, thereby reducing strength. Additional detrimental effects of high fluids content include 1) reduction of reactant concentration in the soil, and 2) entrapment of air in the soil. In the case of immersed strength, with fluids content far below saturation, relatively high humid cure strengths are developed, but immersion in water results in high water absorption with resultant swelling and deterioration of samples. For example, the results in Table II-3 show that none of the MCS samples whose calculated fluids content were below about 11 cc/ 100 gms resisted disintegration on immersion, and samples whose calculated fluids content was below about

10 cc./100 gms. dry soil disintegrated upon immersion after even 7 days humid cure. It is expected that the calculated figure would be more sensitive to relative differences between the samples at the time of immersion. At higher fluids content, where the samples approach saturation, the soil apparently has less tendency to imbibe water, as reflected by the relatively low water pickup and strength decrease upon immersion of samples prepared with high fluids content. The results for VBC, presented in Tables II-4 and II-5, are completely consistent with this explanation.

2. Relation Between Strength and Compacted Density

The results in Tables II-3 and II-4 indicate that there is no correlation between compressive strength and compacted density, which is contrary to previous work. Strengths are generally comparable with previous work, but in many cases strengths are considerably greater than would be expected from the measured densities.

It is believed that the reason for this lack of correlation is that the measured densities are in error due to expansion of samples upon removal from the mold. This problem has not been reported previously, but the present work has involved higher fluids contents than generally used previously, and the problem of expansion is greatest at high fluids content where trapped air cannot readily escape. Since samples always expanded and cracked along planes normal to the axis of compression (due to lateral confinement by the mold), it seems likely that measured densities in many cases are low while compressive strength is not much affected by the cracks normal to the direction of compression.

3. Effect of Capillary Uptake of Water Prior to Immersion

Since it was not possible to mold samples at high water content, two batches of VBC samples (with 1.0% octylamine and 2% phosphoric acid) were prepared in which the water content of the sample was increased via capillary uptake after molding. It was reasoned that introducing water after molding without producing the high pore pressures presumed to be the cause of the strength loss upon immersion would inhibit water uptake when the sample was immersed and result in higher immersed strengths. Accordingly, for these two batches, some samples were placed in 1/16" of water for varying lengths of time during their humid cure. This procedure may be termed "wet humid cure". The data for these two batches are presented in Table II-6. The number of days the samples were subjected to each type of curing condition appears before the letter symbolizing the condition of cure in a column headed "Cure". Thus, "3H4W1I" in this column indicates three days of normal humid cure, 4 days of "wet humid cure", and one day immersion. In all cases the samples

subjected to wet humid cure for any length of time either disintegrated upon immersion or exhibited (1) lower immersed strengths (0-9 psi), (2) lower densities after immersion, and (3) higher water uptake than comparable samples with no wet humid cure. About one-half of the total water uptake and density loss occurred during the wet humid cure. Unfortunately no strength measurements were made before immersion on wet humid cure samples, but it seems reasonable to assume, from the density measurements and water uptake, that these samples would have significantly lower strengths than unimmersed humid cure samples.

From these findings it may be inferred that either capillary uptake without immersion still produces large pore pressures, or that strength loss on immersion may be due largely to the solution of soluble soil components incorporated in the cementing gel. If the latter is true it will be interesting in the future to evaluate the extent of the loss due to the dissolving of soil components upon immersion. In particular, it is recommended that some samples be immersed in small amounts of distilled water and that the soluble components be identified from analysis of the water. In addition, a test might be devised which would subject soil samples which have been successfully stabilized with formulations reported in past work to extended leaching with water under conditions where high pore pressures may not affect the unconfined compressive strength (i. e., laterally confined samples). Such a test would be a measure of the effectiveness of each stabilizing system under field conditions which would tend to dissolve any soluble component incorporated in the cementing gel.

4. Effect of "Free" Sulfuric Acid on Strength

a. Results with MCS

The results in Table II-3 show that "free" sulfuric acid, up to a certain concentration, increases both the humid cure strength and the immersed strength of MCS stabilized with mixtures of phosphate rock and sulfuric acid. For example, for a formulation involving no octylamine and 12.5 cc fluid/ 100 gms soil, MCS samples with 0%, 0.4%, and 1.4% "free" sulfuric acid gave respective one day humid cure strengths of 312 psi, 380 psi, and 425 psi, and respective seven day immersed strengths of 80 psi, 185 psi, and 270 psi. Similar samples with 2.4% "free" sulfuric acid gave very low humid cure and immersed strengths; this may be due in part to the low calculated fluids content of these samples, but it seems clear that this high concentration of "free" sulfuric acid is detrimental to strength. Samples prepared with 0.05% octylamine and 12.5 cc liquids/ 100 gms soil show identical trends; samples with 0%, 1.4%, and 2.4% "free" sulfuric acid gave strengths of, respectively, 235 psi, 505 psi, and 55 psi for seven days

humid cure, and 95 psi, 200 psi, and zero (disintegrated) for seven day immersed cure. The other data in Table II-3, and the data for VBC in Table II-4, show similar trends. While some of these effects may be due in part to differences in apparent (or calculated) fluids content (or volatile content), it seems clear that increasing amounts of "free" sulfuric acid are very beneficial up to about 1.4%, but higher concentrations are detrimental.

These effects are completely consistent with the proposed mechanism of stabilization by phosphoric acid, and can be explained as follows. "Free" sulfuric acid tends to assure more complete conversion of phosphate rock to phosphoric acid, and "free" sulfuric acid also increases the acidity of the soil and may thus promote dissolution of alumina which provides additional cementitious material. The amount of available cementitious material, and the strength, should therefore increase with increasing amounts of "free" sulfuric acid, as observed. According to the proposed mechanism, however, gelation of the cementitious material (and hence strength development) does not occur until most of the acid has been consumed and the pH rises to the point where gelation can occur. "Free" sulfuric acid might therefore be expected to retard strength development (due to the increased acidity and delayed gelation) but increase ultimate strength. With very high concentrations of "free" sulfuric acid (e.g., 2.4%), it might be expected that strength development would be considerably retarded, but the results show that with 2.4% "free" sulfuric acid very little strength is developed after even 28 days humid cure. From this it appears that 2.4% "free" sulfuric acid is such an excess that either gelation is drastically retarded, or else perhaps there is not enough reactive material present in the soil to consume all of the acid even after extremely long cure.

b. Results with VBC

Items 6-9 in Table II-4 show the effect of increasing the excess acid content from 0 to 1.4% in the soil VBC. Apparently items 7 and 8 had water contents such that most of the strength was developed in one day, but 8 with 1.4% excess acid is significantly stronger at both 1 day and 7 days. Item 9 must be near the optimum water content (18.9% fluids at molding) since it increased by 80 psi upon seven days cure. In general 1.4% excess acid will increase 7 day humid cure strengths substantially in samples with the proper water content.

The beneficial effects of excess acid can be seen most clearly, however, in the increase in 7-day immersed strength (30-40 psi) in going from items 6 and 7 (0.0% excess acid) to items 8 and 9 (1.4% excess acid).

Increasing the excess acid content to 2.4% and then 3.4% (1) had little effect on the one day humid cure strength, (2) decreased the seven-day humid cure strength, (3) decreased the seven-day immersed strength, and (4) tended to increase the density.

All of the samples made with 2.4% excess acid (10, 12 and 13) were somewhat irregular. Samples 10 and 13 flocculated during mixing, producing a very sticky though compactable soil. Both of these samples disintegrated upon immersion. The reason for this flocculation is not clear, but it has never been observed at lower acid concentration. Sample 12 was formulated with no octylamine and hence it is not surprising that it disintegrated upon immersion.

Sample 11, with 3.4% excess acid, had a seven day humid cure strength of 255 psi compared to 364 psi for a sample (14) with similar volatiles content and 1.4% excess acid. Likewise the seven-day immersion dropped from 140 psi to 95 psi as acid increased from 1.4% to 3.4%.

In spite of the fact that no reliable data are available for immersed strengths at 2.4% excess acid, the decrease in seven day humid cure strength, coupled with the observation of a substantial decrease in both humid cure strength and immersed strength for samples with 3.4% excess acid, suggests that formulations involving more than 1.4% excess acid do not increase strengths substantially and may even decrease the strength somewhat. Accordingly it is concluded that 1.4% excess acid is at or near the optimum acid content.

These results are completely consistent with the previously mentioned postulated mechanisms.

With the proper excess acid content the phosphate rock-sulfuric acid stabilization system can produce seven day humid cure strengths up to 364 psi and one day humid cure strengths of 140 psi. In the absence of accelerators, however, the one day immersed strength is very low (0-12 psi). Thus, this stabilization system is not a particularly good one for heavy clays and it seems that other stabilizing systems would be more effective for heavy clays such as VBC.

c. Effect of H_2SO_4 in Combination with H_3PO_4 and Other Additives

The results in Table II-7 show that sulfuric acid alone, at a concentration of 2%, provides considerable stabilization of MCS, i.e., 125 psi one day humid cure strength and 45 psi one day immersed strength. This is rather surprising and indicates that part of the effectiveness of "free" sulfuric acid may be due to independent stabilization in addition to the primary stabilization mechanism, i.e.,

perhaps by reaction with alumina in the soil. A series of samples (see Table II-7) was therefore prepared with 0.5% $Al_2(SO_4)_3$, but it can be seen that aluminum salt had no beneficial effect.

The results in Table II-8 indicate that sulfuric acid, added to phosphoric acid and $FeCl_3$, waterproofer, is greatly beneficial for stabilization of VBC. It can be seen that the sulfuric acid decreases short humid cure strengths, as would be expected from the previously mentioned postulated mechanism, but the sulfuric acid greatly improves immersed strengths and longer humid cure strengths, also in agreement with the postulated mechanism.

5. Effectiveness of Octylamine and Armeen D as Waterproofer

a. With MCS

Previous work with MCS has generally indicated that strength loss on immersion after humid cure is relatively slight, and that as little as 0.05% octylamine had a noticeable waterproofing effect. The results of the present investigation, however, indicate that MCS stabilized with mixtures of phosphate rock and sulfuric acid presents serious difficulties as far as achieving adequate immersed strengths after seven days humid cure is concerned, and even more serious as far as immersion after one day humid cure is concerned. The reasons for this difficulty are not clear at present. The soil used in the present work was a different batch of MCS from that used in previous work, but it was obtained from the same location (within a few feet) as previous soil. All of the chemicals used were identical with those used in previous work (i.e., the phosphate rock, sulfuric acid, and octylamine). Humid cure strengths obtained in the present work are comparable with previous work.

The most likely difference seems to be a difference in the soil, either as obtained or as prepared. DTA analysis on the soil as obtained indicated composition essentially identical with the soil used in previous work. Differences in soil processing, however, may be of some significance, although the procedure was the same as that previously used (i.e., drying, grinding to pass 10 mesh, then equilibration to about 7% water content). It has been found, however, that significant amounts of clay remain on the stones which do not pass 10 mesh and which were rejected in processing. While these stones were rejected in previous work also, some slight difference in the water content of the soil at grinding or in the procedure may have resulted in retention of different amounts of this clay, which may account for differences in the soil properties.

At any rate, the results in Table II-3 indicate that both n-octylamine and Armeen D are equally effective as waterproofers for MCS, although in this work neither appears to be very effective as a waterproofer.

b. With VBC

Normal octylamine and Armeen 8 (a commercial grade of n-octylamine) were used as waterproofing agents. One batch was made without octylamine (item 12, Table II-4), but unfortunately both batches of samples with similar acid contents (2.4% excess) flocculated and hence disintegrated upon immersion. Data at other acid contents strongly indicate that these samples would have held together if they had not flocculated upon immersion with strengths around 100 psi and it is concluded that octylamine does waterproof the samples effectively after a seven day humid cure.

It was found that Armeen 8 was equally effective and this material may be more easily available and lower in cost than chemically pure n-octylamine.

In initial work it was observed that addition of the n-octylamine to concentrated sulfuric acid (preparation method A) caused degradation of the n-octylamine, as evidenced by blackish tars which formed. In addition, samples 1, 4 and 5 (Table II-4) all at 0% excess acid either disintegrated or had very low seven day immersed strengths. Samples 2 and 3 at higher acid contents disintegrated upon immersion. While 3 had an abnormally low volatiles content (12.9%), 1 and 2 are comparable to other runs which did not disintegrate.

With preparation B the sulfuric acid was diluted with an equal weight of water prior to addition of the octylamine. When this procedure was used no degradation occurred and Table II-4 shows that seven-day immersed strengths were increased from 3 psi and 17 psi to 68 psi and 80 psi.

Apparently the reaction of the octylamine with the concentrated acid destroys the 8 membered carbon chain of the octylamine. In earlier work (Reference 2) it was established that shorter chain lengths were much less effective as waterproofers. In addition it is possible that the fragmentary particles produced by the organic residue may coat soil particles and inhibit cementation.

It is therefore recommended that concentrated acids be diluted before the addition of octylamine as a soil waterproofing agent.

6. Effect of Sodium Fluosilicate as an Accelerator

Sample 19 in Table II-4 is striking evidence that the strength development of the cure of VBC stabilized with acidic phosphate rock is accelerated by sodium fluosilicate. This sample with humid cure strengths lower than many of the other samples which disintegrated upon one-day immersion showed one-day immersed strength of 72 psi, only a few psi less than the seven-day immersion strengths.

D. Conclusions

1. Limited amounts of "free" H_2SO_4 (in excess of that stoichiometrically required for reaction with phosphate rock) result in great improvements in both humid cure and immersed strengths. About 1.4% "free" H_2SO_4 appears to be optimum; higher concentrations greatly reduce strength. For otherwise identical formulations, use of 1.4% "free" H_2SO_4 results in strength improvements of over 100% in many cases with MCS, and somewhat less improvement with VBC.

2. With MCS, one day humid cure strength of 425 psi and seven day immersed strength of 270 psi can be obtained with a formulation consisting of 3.8% phosphate rock (corresponding to 2% phosphoric acid) and 4.0% H_2SO_4 (corresponding to 1.4% "free" acid above the 2.6% required for stoichiometric reaction with the phosphate rock). With VBC and the same formulation with 1% octylamine added, seven day humid cure strengths of 360 psi and seven day immersed strength of 140 psi can be achieved.

3. Compressive strength of both MCS and VBC was found to be strongly dependent on fluids content at molding. Maximum humid cure strengths are obtained with total fluids content of about 12 cc/100 gms soil with MCS, and with about 19-20 cc/100 gms soil with VBC. Immersed cure strengths increase monotonically with fluids content at molding up to the maximum fluid content levels at which samples could be compacted (i.e., 15 cc/100 gms soil for MCS and 25 cc/100 gms soil for VBC). Strength loss and water absorption on immersion both decrease with increasing fluids content at molding.

4. Both n-octylamine and less expensive Armeen 8 are equally effective waterproofers. They must be added to diluted acid rather than concentrated acid to avoid decomposition. Very little, if any, waterproofer is required with MCS, but waterproofer at levels of about 1% are required with VBC.

5. Two batches of phosphate rock, of approximately the same composition, gave essentially identical results with both soils.

6. Sodium fluosilicate accelerates strength development.

E. Recommendations

1. "Free" sulfuric acid (in excess of that required for stoichiometric reaction with phosphate), at about 1.4% of soil weight, should be used for stabilizing soil with mixtures of phosphate rock and sulfuric acid.

2. Where immersed strength and water resistance are primary considerations, soils should be compacted at as high fluids contents as possible. Further work is recommended to verify this conclusion.

3. Armeen 8, because of its equal effectiveness and lower cost, should be used in place of n-octylamine. The amine should always be added to diluted acid rather than concentrated H_2SO_4 .

4. Mixtures of phosphate rock and sulfuric acid, with about 1.4% "free" H_2SO_4 , appear to be very promising for stabilizing MCS, and it is recommended that this system be further investigated for stabilization of lean soils. Stabilization of heavy clays with acidic phosphorus compounds does not appear to be as attractive as other systems (e.g., lime), and it is recommended that other inorganic systems should be investigated.

F. References

1. "Soil Stabilization by Chemical Methods , Final Report, Phase IX", November, 1957
2. "Soil Stabilization by Chemical Methods, Final Report, Phase X", November, 1958
3. "Soil Stabilization by Chemical Methods, Final Report, Phase XI", November, 1959
4. "Soil Stabilization by Chemical Methods, Final Report, Phase XII", November, 1960
5. Personal Communication, U. S. Army Corps of Engineers, Vicksburg, Mississippi, May, 1961

TABLE II-1
PROPERTIES OF SOILS INVESTIGATED

Properties	Mass. Clayey Silt	Vicksburg Buckshot Clay
<u>Textural Composition¹ % by weight:</u>		
Sand, 0.06 mm to 2 mm	47	0
Silt, 0.002 mm to 0.06 mm	41	65
Clay, < 0.002 mm	12	35
<u>Physical Properties:</u>		
Liquid limit, %	20	63-67
Plastic limit, %	14	27-28
Plasticity index, %	6	35-40
Specific gravity 20°C/ 20°C ₃	2.7	2.67
Maximum dry density ² lb/ ft ³	122.0	105.0
Optimum water content, %	13.3	22.0
<u>Chemical Properties:</u>		
Organic matter, % by weight	--	1.1
pH	--	4.6
Total soluble salts, as m. eq. NaCl/ 100 gm	--	0.3
Soluble sulfates, m. eq. NaCl/ 100 gm	--	--
Carbonates, % by weight	--	--
<u>Mineralogical Compositions:³</u>		
Clay composition, %	30	50
Illite:montmorillonoid:clay-chlorite	1:0:1	1:1:0
Chlorite, nonclay, %	none	none
Calcite, %	none	none
Free iron oxide, % Fe ₂ O ₃	2.9	1.9
Gypsum, %	none	none
<u>Source:</u>	Mass.	Miss.

¹ M. I. T. Classification System

² Harvard Miniature Compaction, 40-lb. tamper, 3 layers, 25 blows/ layer

³ For -0.074 mm fraction

TABLE II-2
PHOSPHATE ROCK PROPERTIES

	Batch I	Batch II
Particle Size		
Through 60 mesh	93.6%	91.2%
Through 100 mesh	75%	76.8%
Through 200 mesh	63%	52.8%
Moisture Content	1.26%	1.06%
Bone Phosphate of Lime	71.41%	74.86%
Total P₂O₅	32.27%	33.90%
Combined Flourine	2.72-4.08%	--

TABLE II-7
STABILIZATION OF M-21 WITH MIXTURES OF H_3PO_4 AND H_2SO_4

Days Humid Cure	1						7						Fluids Content at Molding (Measured), cc/100 gms dry solids
	% Vol. at mold.	C. S. pcf	% Vol. at test.	D. D. pcf	% Vol. at mold.	C. S. pcf	% Vol. at test.	D. D. pcf	% Vol. at mold.	C. S. pcf	% Vol. at test.	D. D. pcf	
2% H_3PO_4 , 2% H_2SO_4	9.7	165±15	9.7	133	10.0	115±5	11.1	131.75	10.5	180	12.9	131.5	15
2% H_2SO_4	9.4	125±5	9.5	134.5	11.0	45±5	11.8	133	11.1	50	12.4	131.75	15
2% H_3PO_4 , 2% H_2SO_4 , 0.5% aluminum sulfate	10.13	110	10.15	134	11.55	95±15	12.2	131	11.7	110	12.75	131.5	15
3.82% phosphate rock, 2.6% H_2SO_4	11.3	205±5	11.3	129	10.8	100	12.5	129	11.0	240	11.3	129	15
3.82% phosphate rock, 2.6% H_3PO_4	9.4	205±15	9.35	126.5	9.5	75±5	13.45	126	9.6	245±25	92	176.5	10

TABLE II-8
STABILIZATION OF VBC WITH MIXTURES OF H_3PO_4 AND H_2SO_4

Days	Humid	Cure	1	1	7	7
Days	Immersion	0	0	0	0	1
	C. S. psi	% Vol.	D. D. pcf	C. S. psi	D. D. pcf	C. S. psi
				Vol.	Vol.	Vol.
2% H_3PO_4 , 2% H_2SO_4						
1.6% $FeCl_3$	115±5	20.7	109	65±5	25.5	106.5
2% $H_3PO_4^1$, 1.6% $FeCl_3$	220±11	15.7	112.4	Samples held together but could not be tested		
2% H_3PO_4 , 1.6% $AlCl_3$	360±10	17.2	114.0	32±1	--	--

¹ Reference 4, Table III-4

III. SOIL STABILIZATION WITH LIME

A. Introduction

Research during the past few years has shown that lime is a good stabilizer for soils of high plasticity, such as clays. The most recent work at the M. I. T. Soil Stabilization Laboratory (Reference 1) was directed mainly to the use of calcium oxide rather than calcium hydroxide due to its cost advantage. The main conclusions reached during the previous research period (Ref. 1) are: (1) calcium oxide is an effective stabilizing agent for VBC; (2) magnesium sulfate considerably improves the strength of calcium oxide stabilized VBC; and (3) calcium oxide by itself will not stabilize M-21.

Research during this contract period (October '60 - October '61) aimed primarily at: (1) clarifying the soil-lime and soil-lime-additive stabilization mechanisms; (2) investigating the effectiveness of: (a) magnesium sulfate in conjunction with other secondary additives, especially sodium metasilicate, and (b) secondary additives other than magnesium sulfate, such as the sulfates of zinc, nickel, copper, aluminum and the inorganic compounds; (3) the stabilization of plastic or "fat" soils other than VBC. Specifically two other soils were used--Houston Black Clay (HBC) and Siburia Soft Shale (SSS).

B. Experimental

1. Materials

The soils used in this investigation were Vicksburg Buckshot Clay (VBC), Houston Black Clay (HBC), and Siburia Soft Shale (SSS). The properties of VBC are given in Table II-1. The VBC used in this investigation was obtained from a different location than the soil used in previous work. However the Atterberg limits and DTA measurements were found to be the same for both soils. Complete quantitative data on the properties of the other two soils are lacking, and hence only qualitative comparisons are made in the discussion of results.

The following reagent grade chemicals were used: calcium oxide (CaO), calcium hydroxide ($\text{Ca}(\text{OH})_2$), hydrous and anhydrous magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and MgSO_4), basic magnesium carbonate ($4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$), magnesium fluoride (MgF_2), magnesium oxide (MgO), ammonium chloride (NH_4Cl), sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$), zinc sulfate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$), nickel sulfate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$), copper sulfate (CuSO_4), and aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$).

2. Procedure

Preparation and testing of samples were the same as those described in previous reports. VBC was first equilibrated for 24 hours with approximately half of the molding water content (8 to 10%). The other soils were used directly. All additives were preblended and the mixture added in powder form to the soil and mixed in a finger prong mixer for a few minutes. The additional molding water was added subsequently while mixing. Samples were then prepared by two-end static compaction in a Harvard Mineature mold at approximately 1000 psi. The samples were cured at 100% relative humidity for one, seven and 28 days, then immersed in water for one day, and finally tested in unconfined compression. The tested samples were dried at 110°C for at least 24 hours and weighed to determine water content. Samples were also weighed and measured after molding, curing and immersion.

C. Results

I. Results of Work with VBC Only

1. Results Obtained with the New Batch of VBC

During the present investigation, while working with a new batch of VBC and CaO, it was found that the results obtained with a previous shipment of soil (Reference 1) could not be duplicated. The following discrepancies were observed (Table III-1):

a. Optimum Water Content. The optimum water content, defined as the water content at which maximum strength is obtained, is considerably lower for the "new" soil (17% for the new compared to 22% for the old soil).

b. Density. The densities obtained with the new soil are considerably higher than those obtained with the old soil (3 to 5 pounds per cubic foot) at the optimum water content of each soil and for the same compactive effort (1000 psi).

c. Strength. With 5% CaO, the strengths obtained were lower for the new soil in spite of higher densities; seven day immersed strengths were 215 psi for the old soil and 150 psi for the new. With 6.6% Ca(OH)₂, the strengths obtained with the new soil were higher; seven day immersed strengths were 50 psi for the old and 165 psi for the new soil. Furthermore the results obtained during the previous period (Reference 1) have shown that stabilization with CaO gives greater

strengths than Ca(OH)_2 . It was also observed that Ca(OH)_2 gave lower dry densities than CaO . Results obtained in the present investigation, however, with the new batch of soil, have shown no such trends. The strength obtained with CaO and Ca(OH)_2 at equivalent calcium concentrations (5% CaO and 6.6% Ca(OH)_2) were approximately equal; one day and seven day immersed strengths were 125 psi with CaO and 150 psi with Ca(OH)_2 , and 150 psi with CaO and 165 psi with Ca(OH)_2 respectively. The dry densities obtained with both CaO and Ca(OH)_2 were also approximately equal, but considerably lower than the density of the soil alone without any additive and at approximately similar water contents (112 pcf for the untreated soil compared with approximately 104 pcf for the treated soil).

d. Effect of Magnesium Sulfate. Magnesium sulfate produced an increased strength with both CaO and Ca(OH)_2 . The improvement in strength of the CaO stabilized soil, caused by the magnesium sulfate, was approximately the same in the new and old soil, 80% for the new soil and 84% for the old (after 7 days humid cure and one day immersion). However, in the case of Ca(OH)_2 stabilized soil the strength increase due to magnesium sulfate was much greater in the old soil than in the new one, 170% for the old and 20% for the new soil (after 7 days humid cure and one day immersion).

A similar situation was encountered previously (Reference 3) in the stabilization of VBC with phosphoric acid. The difference in behavior of the two batches of soil were attributed to the "histories" of the soils. The batch which gave lower densities was received wet and had been dried for only a short period of time, while the soil which gave high densities, and which had a lower optimum water content, had been left at ambient conditions for several years. It was therefore suggested that the long drying period might have caused some irreversible shrinkage of the clay lattice, thus lowering the optimum water content. To test this hypothesis, the history of the "low density" soil was roughly simulated with the "high density" soil by oven drying it at 110°C . The soil was then re-equilibrated with water and subsequently stabilized under similar conditions and tested. The results showed that the properties of the soil were modified as expected.

The discrepancies in the results obtained in this investigation may therefore be due to the different histories of the old and new soils. It was not possible to simulate the properties of the soils by oven drying because the old soil was not available, but oven drying of the new soil had no effect on its properties, as expected from the above hypothesis.

2. Optimum Ratio of Calcium Oxide to Magnesium Sulfate, and Effectiveness of Anhydrous Magnesium Sulfate

In all previous work, the ratio of calcium oxide to magnesium sulfate was kept at four parts by weight CaO to one part by weight $MgSO_4 \cdot 7H_2O$. Different weight ratios were tried in this investigation by varying the amount of $MgSO_4 \cdot 7H_2O$ and keeping the CaO concentration fixed at 5%. The results for VBC (Table III-2) show that the optimum weight ratio of CaO to $MgSO_4 \cdot 7H_2O$ is four to one. Higher proportions of magnesium sulfate gave lower strengths even with samples that contained the same amount of free CaO as those with a four to one ratio.

Anhydrous magnesium sulfate was also tried, at a concentration equivalent to the optimum of the heptahydrate (0.61% by weight). The results obtained with the anhydrous form are entirely comparable with those of the heptahydrate (seven day immersed strengths are 275 psi for the anhydrous and 270 psi for the heptahydrate) at equivalent Ca concentrations.

3. Relative Effectiveness of Additives

Several compounds other than $MgSO_4$ were investigated as secondary additives for the stabilization of VBC with 5% CaO (Table III-3a). These compounds were:

Basic magnesium carbonate ($4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$)
Magnesium fluoride (MgF_2)
Magnesium oxide (MgO)
Ammonium chloride (NH_4Cl)
Sodium metasilicate ($Na_2SiO_3 \cdot 9H_2O$), with and without magnesium sulfate ($MgSO_4 \cdot 7H_2O$)
Zinc Sulfate* ($ZnSO_4 \cdot 7H_2O$)
Nickel Sulfate* ($NiSO_4 \cdot 6H_2O$)
Copper Sulfate* ($CuSO_4$)
Aluminum Sulfate* ($Al_2(SO_4)_3$)

*With and without $Na_2SiO_3 \cdot 9H_2O$

The three magnesium compounds were used at Mg concentrations equivalent to the optimum for magnesium sulfate, i.e., 1.25% $MgSO_4 \cdot 7H_2O$ on dry soil. The results obtained with these three compounds are comparable for all cures (Table III-3a). One day immersed strengths were 115 psi, 125 psi, and 110 psi respectively, and the

seven day immersed strengths were 145 psi, 130 psi, and 160 psi respectively. These strengths are approximately the same as those obtained with 5% CaO alone, and generally slightly lower (10-20 psi).

The amount of ammonium chloride used was one quarter the stoichiometric amount required for combination with CaO. The results obtained with this compound were better than those obtained with the above mentioned magnesium compounds and with 5% CaO alone. Seven day immersed strength was 210 psi compared with 150 psi for 5% CaO alone.

Sodium metasilicate was used at two concentration levels when used alone, corresponding to approximately 0.5N and 1.0N solutions in the molding water. It was also used in conjunction with $MgSO_4$, $ZnSO_4$, $NiSO_4$, $CuSO_4$ and $Al_2(SO_4)_3$ at a concentration of 0.5 N in molding water. These concentrations were chosen on the basis of a previous investigation (Reference 4) of the effect of various sodium additives on soils stabilized with hydrated lime ($Ca(OH)_2$). The results obtained in the above investigation showed that, whereas metasilicate was the most effective additive for most soils, for VBC it was only moderately effective. It also showed that with VBC the maximum strength was obtained with 0.5 N metasilicate in molding water.

The present investigation of the effect of sodium metasilicate with 5% CaO on the stabilization of VBC has shown that (Table III-3): (1) the optimum water content for this system is higher than for other CaO systems (samples prepared with 17% water content disintegrated on humid cure); (2) the optimum metasilicate concentration seems to be between 0.5 N and 1.0 N in the molding water, the strengths increasing only slightly with increasing metasilicate concentrations; (3) the strengths obtained by addition of the metasilicate are greater than those with 5% CaO alone but lower than those obtained with 5% CaO and 1.25% $MgSO_4$; that is, the strengths are intermediate between those of CaO alone and CaO plus $MgSO_4$. One day immersed strength with 0.5 N metasilicate is 170 compared with 125 psi with CaO alone and 235 psi with CaO plus $MgSO_4$; (4) the strengths obtained with CaO in combination with both metasilicate and magnesium sulfate, however, are greater than those obtained with CaO plus magnesium sulfate (1.25% $MgSO_4 \cdot 7H_2O$ used in both cases). One day immersed strengths were 265 psi with 0.5 N metasilicate and 235 psi with CaO plus $MgSO_4$.

The following list of one and seven day immersed strengths, arranged in order of increasing strengths, shows the relative effectiveness of metasilicate:

	One Day Strength psi	% Improve- ment over CaO	Seven Day Strength psi	% Improve- ment over CaO
5% CaO	125		150	
5% CaO + Na ₂ SiO ₃ (0.5 N)	170	36	200	33
5% CaO + MgSO ₄ (1.25%)	235	88	270	80
5% CaO + Na ₂ SiO ₃ (0.5 N) + MgSO ₄ (1.25%)	265	112	330	120

Table III-3b lists the results obtained with the last four compounds, namely ZnSO₄, NiSO₄, CuSO₄, and Al₂(SO₄)₃, both alone and with Na₂SiO₃. These compounds were used at concentrations equivalent to 1.25% MgSO₄ · 7H₂O. The best results were obtained with ZnSO₄ and NiSO₄ which caused an improvement in strength comparable to that produced by MgSO₄. One day immersed strengths are 200 psi for ZnSO₄ and 170 psi for NiSO₄ compared to 235 psi for MgSO₄. The seven day immersed strengths are 230 psi for ZnSO₄ and 275 psi for NiSO₄ compared to 270 psi for MgSO₄, indicating that these additives are equally effective. In the case of CuSO₄ the one day immersed strengths are the same as those with NiSO₄, but the seven day cures are considerably lower (180 psi). Al₂(SO₄)₃ seems to have no effect, no improvement in strength being obtained over that of VBC + CaO alone.

The same trend in relative effectiveness of these additives is found when Na₂SiO₃ is incorporated. Best results were obtained with the sulfates of Zn and Ni, and lower strengths were obtained with CuSO₄. However, only a very slight improvement in strength was obtained when Na₂SiO₃ was used with these salts compared to a substantial increase in the case of MgSO₄ + Na₂SiO₃. One day immersed strengths are 210 psi for ZnSO₄, 190 psi for NiSO₄, and 180 psi for CuSO₄ compared to 265 psi for MgSO₄. Seven day immersed strengths are 205 psi, 280 psi, and 200 psi respectively compared to 330 psi for MgSO₄.

II. Results with Other Soils

1. Houston Black Clay (HBC)

A comparison of the physical properties of this soil with VBC is as follows:

	VBC	HBC
Liquid limit, %	63-67	71
Plastic limit, %	27-28	27
Plasticity index	35-40	44
Specific gravity 20°C / 20°C	2.67	2.71
Max. dry density, lb/ ft³	105.0	105.0
Optimum water content, %	22.0	20.0

From the above it can be seen that HBC is very similar to VBC in its physical properties and seems to be even more plastic than VBC.

Even though the exact mineralogical composition of the HBC used is not known, comparison of other Houston Black Clays with VBC reveals that the montmorillonoid content of HBC is higher than that of VBC.

Table III-4 lists the results obtained by stabilizing this soil with CaO. Various percentages of CaO were used ranging from one per cent to seven per cent. The secondary additives that proved beneficial with VBC, namely $MgSO_4$, Na_2SiO_3 , $ZnSO_4$, and $NiSO_4$ were also used with this soil.

The results indicate that maximum strength is attained with between two and three per cent CaO, strengths falling off fairly rapidly above and below this concentration. The one day immersed strengths with 2% and 5% CaO are 315 psi and 260 psi respectively and drop to 100 psi with 7% CaO. A similar trend is followed with the seven day immersed strengths; 2%, 5%, and 7% CaO strengths are 525 psi, 410 psi, and 185 psi respectively. The maximum strengths attained with VBC are with 5% CaO, above which concentration the strengths drop only slightly (Reference 1).

A considerable improvement in strength is obtained with both $MgSO_4$ and Na_2SiO_3 , the increase in strength being about the same for both compounds. One day immersed strengths are 390 psi for $MgSO_4$ and 375 psi for Na_2SiO_3 , compared with 260 psi for HBC + 5% CaO alone. The seven day immersed strengths are 470 psi, 475 psi, and 410 psi respectively. Maximum strength is obtained, however, when

$MgSO_4$ and Na_2SiO_3 are used together (505 psi and 735 psi after one day and seven days respectively).

Of the two other additives used, $NiSO_4$ produces an increase in strength, while $ZnSO_4$ seems to be detrimental. The strengths obtained with $NiSO_4$ are comparable to those obtained with $MgSO_4$ (seven day immersed strength is 495 psi).

It is interesting to note here that, unlike the other soils used, HBC in many instances gives higher immersed strengths than those obtained on humid cure without subsequent 24 hours immersion.

2. Siburua Soft Shale (SSS)

The results on the stabilization of this soil with CaO are given in Table III-5. Although detailed data on the physical properties and mineralogical composition of this soil are not available, examination of the data (especially mineralogical composition) of other soils from the same area (samples from Siburua) indicate that this soil differs from the other two (HBC and VBC) in that these soils have a much lower montmorillonoid content (almost one third that of HBC) and a relatively high organic matter content compared with VBC which has about 0.1%.

The results indicate that CaO produces strengths higher than 200 psi after one day humid cure. After immersion, however, the samples lose their strength. For all practical purposes, therefore, it may be concluded that CaO does not satisfactorily stabilize this soil. Furthermore, the results indicate that the additives $MgSO_4$, Na_2SiO_3 , $ZnSO_4$, and $NiSO_4$ produce no improvement in strength.

III. Proposed Soil-lime-additive Stabilization Mechanism

Soil-lime stabilization is thought to proceed (Reference 5) as follows: the addition of lime causes an immediate increase in pH of the molding water, caused by the hydration of calcium oxide. The high pH in the pore water solubilizes part of the soil silicates and/or aluminates, which in turn react with the calcium ions giving a cementitious calcium silicate gel.

Soil-lime stabilization may thus be improved by incorporating substances which will: (1) produce greater amounts of reactive silica, (2) control the rate of reaction of calcium with silica to obtain a more uniform distribution of calcium silicate throughout the soil (Reference 5), or (3) produce silicate or other gels with properties superior to calcium silicate.

Greater amounts of silica may be produced by either an increase in pH of the pore water, which will solubilize greater amounts of silica from the soil particles, or by addition of water soluble metasilicate. The data show that addition of metasilicate with the CaO produces greater strengths than with CaO alone. This indicates that not all of the CaO reacts with the soil silica and the remaining unreacted CaO may thus react with the added silicate to provide additional cementitious calcium silicate gel. It is postulated that the reaction of silica with the Ca is controlled as follows: the lime reacts with the sodium metasilicate to form calcium silicate and sodium hydroxide (causticization),



which greatly depresses the calcium concentration (due to the insolubility of CaX) and at the same time elevates the pH significantly. The pH elevation accelerates attack of soil silica and formation of additional sodium silicate which is free to diffuse through the pore fluid. Eventually, however, neutralization of the free alkali by reaction with the soil reduces the pH, which permits the calcium ions to go into solution and react with the uniformly distributed alkali silicate.

In an effort to explain the improvement in strength caused by ~ agnesium sulfate, a number of pH measurements were made on aqueous solutions of CaO and CaO plus MgSO_4 , to determine whether magnesium sulfate has any effect on pH. Measurements were made with a Beckman pH meter, both manually and by means of a recorder. The concentrations varied from dilute solution (1 gm CaO in 100 cc water) to slurries (20 gm CaO in 100 cc water) comparable to the concentrations developed in the samples. When magnesium sulfate was used it was dissolved in water before adding to the CaO. The measurements indicated a peak pH value of approximately 12.4 with the concentrated solutions, which dropped off to approximately 12.1 in a few seconds. When magnesium sulfate was used the peak pH value was the same and there was no indication that the subsequent drop in pH was greater than in the case of CaO alone. From these results one may conclude that magnesium sulfate has no effect on the pH of the pore water of the treated soil samples.

The improvement in strength caused by the addition of magnesium sulfate was previously thought to be due to formation of a Ca-Mg-Silicate gel (Reference 1) with properties superior to the Ca-Silicate gel. The results with sodium metasilicate and the three salts MgSO_4 , ZnSO_4 , and NiSO_4 seem to confirm this view. The strengths obtained when these salts are used in conjunction with the Na_2SiO_3 are greater than with the silicate alone and greater than with MgSO_4 without Na_2SiO_3 .

This is true for both VBC and HBC. These observations suggest the formation of a Ca-M-Silicate gel (where M is Mg, Zn, or Ni) with properties superior to Ca-Silicate.

The other magnesium compounds, namely magnesium carbonate, fluoride, and oxide, did not produce any improvement in strength with VBC. The results were approximately the same as those with CaO alone.

The extremely low solubilities of these compounds, and their consequent inability to react rapidly with lime, may account for their ineffectiveness.

It should also be pointed out that magnesium, zinc, and nickel form stable, insoluble basic salts of strong acids, and that, in the case of magnesium particularly, these basic salts (e.g., the oxysulfate and oxychloride) are powerful cementing agents in their own right. The possibility cannot be excluded, therefore, that the observed significant beneficial action of the sulfates of magnesium, zinc, and nickel on lime stabilization may result from the formation of oxy-sulfates which, in conjunction with hydrous calcium silicate, serve to produce a superior cementitious product.

Nothing has been said in the proposed mechanism outlined above to justify the difference in the results obtained with various soils. The soils investigated so far at the M. I. T. Soil Stabilization Laboratory are: VBC, HBC, SSS, and M-21. Table III-6 lists the main results obtained with these soils. The main observations from Table III-6 are:

1.) CaO alone is a good stabilizing agent for VBC and HBC, a mediocre one with SSS, and ineffective with M-21. Furthermore the strengths obtained with HBC are much greater than those with VBC (in many cases more than twice as high). The reason for this difference in results is believed due to the amount of reactive silica in the soils. Montmorillonoid being the mineral supplying the reactive form of silica (Reference 6), it is expected that strength development will be related to the amount of montmorillonoid in the soil. The results obtained with these soils seem to confirm this hypothesis. CaO does not stabilize M-21, stabilized SSS to a very limited extent, and is a good stabilizer for HBC and VBC.

2.) The improvements in strength caused by the use of additives are as follows:

Per Cent Increase in Strength Over Soil + 5% CaO

Soil	MgSO ₄		Na ₂ SiO ₃		MgSO ₄ + Na ₂ SiO ₃		ZnSO ₄		NiSO ₄	
	1HII	7HII	1HII	7HII	1HII	7HII	1HII	7HII	1HII	7HII
VBC	88	80	36	33	116	113	60	53	36	83
HBC	50	11	44	15	94	79	-21	-44	73	21
SSS	-50	-11	-25	-11	n. a.	n. a.	-11	-11	-12	-11
M-21	0 \rightarrow 100 psi	0 \rightarrow 125 psi	n. a.	n. a.	n. a.	n. a.	n. a.	n. a.	n. a.	n. a.

1HII: One day humid followed by one day immersion

7HII: Seven day humid followed by one day immersion

n. a.: Not available

Some fairly broad conclusions may be reached from the above table: a) additives generally produce a greater improvement in strength with VBC than with HBC, b) additives are detrimental in the case of SSS, and c) MgSO₄ causes a substantial improvement with M-21.

It is deduced from these observations that magnesium sulfate and sodium metasilicate are particularly beneficial additives for quicklime stabilization of high-montmorillonoid, high-plasticity soils, and that zinc or nickel sulfates are generally comparable with magnesium sulfate in their action. Earlier work has shown that lime-magnesium sulfate and lime-sodium silicate are effective stabilizing systems for low plasticity soils such as M-21, whence it is apparent that the ternary system CaO-MgSO₄-metasilicate is a remarkably broad-spectrum soil stabilizer. The inefficacy of this system with SSS is anomalous, and may be due to the peculiarly high plasticity of this soil despite its low montmorillonoid content. Inasmuch, however, as this soil is virtually unresponsive to lime treatment alone, and since it is known to possess relatively high organic matter content, it seems not unlikely that interference by the organic components with the cementation reactions may be responsible for the poor stabilization. This possibility is strongly supported by the well-known fact that high-organic soils respond poorly to stabilization by portland cement.

D. Conclusions

1. Optimum water content and dry density of lime-stabilized VBC varies according to the previous history of the soil. Soils that have been dried for a prolonged period of time give lower optimum water contents (4 to 5 per cent lower) and higher densities (3 to 5 pcf) than the soils which have been dried for a short period of time.

2. Calcium oxide and calcium hydroxide stabilize VBC equally effectively (seven day immersed strengths are 150 psi and 165 psi respectively).

3. The dry densities obtained with calcium oxide and calcium hydroxide are about equal, but considerably lower than the density of the untreated soil (seven day cure densities were 103.4 pcf for CaO, 104.4 pcf for Ca(OH)₂, and 112 pcf for the untreated soil).

4. Magnesium sulfate ($MgSO_4 \cdot 7H_2O$) increases the compressive strength of both the CaO and Ca(OH)₂ stabilized VBC. The improvement in strength however is much greater with CaO than with Ca(OH)₂ (80% improvement with CaO, compared with 20% improvement with Ca(OH)₂ for the seven day immersed strengths).

5. Anhydrous magnesium sulfate is as effective as the heptahydrate form (seven day immersed strengths are 270 psi with the heptahydrate and 275 psi with the anhydrous magnesium sulfate) when used at the optimum calcium to magnesium sulfate ratio.

6. The optimum weight ratio of calcium oxide to magnesium sulfate ($MgSO_4 \cdot 7H_2O$) is four to one.

7. Hydration of calcium oxide gives a peak pH of 12.4 which drops to 12.1 in a few seconds. Magnesium sulfate has no effect on either the peak pH or the drop in pH.

8. Calcium oxide is a good stabilizer for HBC, producing higher strengths than with VBC (one day immersed strengths are 125 psi for VBC and 260 psi for HBC). It does not, however, stabilize M-21 or SSS.

9. Magnesium sulfate improves the strength of CaO-stabilized HBC, VBC and M-21, but not of SSS.

10. Metasilicate improves the strength of CaO-stabilized HBC and VBC, but not of SSS.

11. Zinc sulfate, nickel sulfate, and copper sulfate improve the strength of CaO-stabilized VBC as much as magnesium sulfate. However, when metasilicate is used together with these salts, magnesium sulfate produces the greatest effect.

12. Magnesium sulfate and nickel sulfate improve the strength of CaO-stabilized HBC, but zinc sulfate does not. The improvement is greatest when metasilicate is used together with these salts.

13. The effectiveness of CaO as a stabilizing agent appears to vary with the amount of montmorillonoid (supplying the reactive silica) in the soil.

14. The poor response of SSS to lime stabilization may be due to its high organic matter content.

E. References

1. "Soil Stabilization Final Report", Phase XII, November, 1960.
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5. T. W. Lambe, A. S. Michaels and Z. C. Moh, "Improvement of Soil-Cement with Alkali Metal Compounds", preprint of paper presented before the 38th Annual Meeting of the Highway Research Board, Washington, D. C., January, 1959.
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TABLE III-1
STABILIZATION OF NEW AND OLD VBC WITH LIME

Days	Humid Cure	1		7		28				
		0	1	1	1	1	1	1	1	1
Days	Immersion	C. S. ¹	D. D. ²	Vols. ³	C. S.	D. D.	Vols.	C. S.	D. D.	Vols.
		psi	pcf	%	psi	pcf	pcf	psi	pcf	%
VBC(Old)+ CaO 5%	330	102.0	22.7	195±5	100.2	24.7	215±5	100.8	24.0	265±5
VBC(New)+ CaO 5%	190±10	99.4	23.7	--	--	--	190	98.5	23.5	--
VBC(New)+ CaO 5%	190±5	100.0	22.5	120±10	98.9	25.1	140±5	99.9	23.6	165±5
VBC(New)+ CaO 5%	220±10	101.1	22.0	--	--	--	195±5	100.9	22.8	--
VBC(New)+ CaO 5%	270±5	104.9	17.0	125±5	104.5	21.4	150±5	103.4	22.3	200
at optimum water content										
VBC (new dried) + CaO 5%	210±5	105.2	16.5	105±5	104.7	20.8	100±5	104.8	21.2	--
VBC(Old)+ CaO 5% + MgSO ₄ 1. 25% ⁴	395±15	102.0	22.9	210	99.0	25.4	395±5	101.0	23.7	410±5
VBC(New)+ CaO 5% + MgSO ₄ 1. 25%	360±10	106.2	16.7	235±15	103.6	20.6	270±5	105.6	20.9	320
VBC(Old)+ Ca(OH) ₂ 6.6% ⁵	97.8	20.0	80±10	98.4	24.2	50±10	97.5	24.6	--	104.4
VBC(Old)+ Ca(OH) ₂ 6.6% + MgSO ₄ 1. 25%	225±5	97.3	24.7	95±5	97.7	24.8	135±15	97.6	25.3	--
VBC(New)+ Ca(OH) ₂ 6.6%	270±15	103.9	16.7	150±10	103.8	21.1	165±5	104.4	21.3	--
VBC(New)+ Ca(OH) ₂ 6.6% + MgSO ₄ 1. 25%	290±5	103.9	16.8	165±5	102.4	23.3	200±5	103.0	21.9	240
Density of VBC alone at optimum water content --	112.2	16.5	Disintegrated	--	112.6 ⁶	16.2	--	--	--	--

¹ C. S. is actual compressive strength in psi

² D. D. is dry density at test, i.e., pounds dry solids per cubic foot

³ Per cent volatiles at test, i.e., weight lost upon drying as a percentage of dry solids weight

⁴ Hydrated form of magnesium sulfate (MgSO₄·7H₂O) used, unless otherwise indicated

⁵ 6.6% Ca(OH)₂ is equivalent to 5.0% CaO

⁶ Seven days humid cure only, not immersed

TABLE III-2
OPTIMUM MAGNESIUM SULFATE TO CALCIUM OXIDE RATIO
AND EFFECTIVENESS OF ANHYDROUS MAGNESIUM SULFATE

Days Humid Cure	1					7					28				
	C. S. ¹	D. D. ²	Vols. ³	C. S.	D. D.	Vols.	C. S.	D. D. ²	Vols.	C. S.	D. D.	Vols.	C. S.	D. D.	Vols.
Days Immersion	psi	pcf	%	psi	pcf	%	psi	pcf	%	psi	pcf	%	psi	pcf	%
VBC(New) + CaO 5%															
+ 0.50% MgSO ₄	360±10	104.8	16.9	210±5	104.7	21.7	215±5	104.5	21.0	290	104.8	20.3			
VBC(New) + CaO 5%															
+ 1.00% MgSO ₄	380±5	105.1	17.0	205±5	104.4	21.4	250±10	104.3	21.6	300	105.2	20.5			
VBC(New) + CaO 5%															
+ 1.25% MgSO ₄	360±10	106.2	16.7	235±15	103.6	20.6	270±5	105.6	20.9	320	104.4	21.5			
VBC(New) + CaO 5%															
+ 1.50% MgSO ₄	405±5	105.1	16.0	195±5	103.2	22.3	270±5	103.8	22.7	275	104.5	21.2			
VBC(New) + CaO 5%															
+ 2.00% MgSO ₄	395±5	104.5	16.9	145±5	101.1	23.6	245±15	104.2	20.0	325	104.1	21.2			
VBC(New) + CaO 5.17%															
+ 2.00% MgSO ₄	360±10	105.0	15.0	105	100.5	25.0	265±5	103.9	20.7	240	103.2	21.1			
VBC(New) + CaO 5.17%															
+ 2.00% MgSO ₄	370±5	105.1	16.1	135±5	101.8	23.0	275±10	104.5	21.5	300	104.0	21.9			
VBC(New) + CaO 5%															
+ 0.61% MgSO ₄ (Anh.) ⁵	390±5	105.3	16.5	245±5	104.0	22.1	275±5	104.7	21.5	275	104.9	21.1			

¹ C. S. is actual compressive strength in psi

² D. D. is dry density at test, i. e., pounds dry solids per cubic foot

³ Per cent volatiles at test, i. e., weight lost upon drying as a percentage of dry solids weight

⁵ 1.17% CaO and 2.0% MgSO₄·7H₂O provides the same amount of free CaO as with 5.0% CaO and 1.25% MgSO₄·7H₂O

0.61% MgSO₄ is equivalent to 1.25% MgSO₄·7H₂O

TABLE III-3a
STABILIZATION OF VBC (NEW) WITH 5% CaO AND SECONDARY ADDITIVES

Days Immersion	Days Humid Cure	1			7			28			
		C. S. ¹ psi	D. D. ² pcf	Vols. ³ %	C. S. psi	D. D. pcf	Vols. ³ %	C. S. psi	D. D. pcf	Vols. ³ %	
0	0	104.9	17.0	125±5	104.5	21.4	150±5	103.4	22.3	200	
1	1	235±15	103.6	20.6	270±5	105.6	20.9	320	104.4	21.5	
2	2	65	Cracked		110	104.3	21.0	140	103.2	22.2	
3	3	104.6	14.0	Disintegrated	110	104.7	21.0	--	--	--	
4	4	103.9	15.7	115±5	103.4	22.0	145±5	103.8	21.2	150	
5	5	103.9	15.7	125±5	103.8	21.9	130±5	104.0	21.1	150	
6	6	103.8	15.5	110±5	103.4	22.0	160±10	104.0	21.6	140	
7	7	106.2	20.5	140±5	104.8	21.9	210±15	104.6	22.5	--	
8	8	Disintegrated on humid cure								--	
9	9	90±10	103.8	22.7	145±5	103.6	22.2	--	--	--	
10	10	170±20	103.2	23.0	200±5	103.5	21.9	200	103.5	22.1	
11	11	105.7	18.5								
12	12	350±5									
13	13	Na ₂ SiO ₃ 1.30% ⁷									
14	14	Na ₂ SiO ₃ 1.30%									
15	15	Na ₂ SiO ₃ 1.30%									
16	16	Na ₂ SiO ₃ 1.3% MgSO ₄ ⁸									
17	17	1.25%	475±30	20.3	265±5	103.7	22.7	330±5	102.8	23.1	--
18	18	Na ₂ SiO ₃ 2.00%	265±20	103.6	115±5	102.9	23.2	150±15	103.3	22.7	--
19	19	Na ₂ SiO ₃ 2.00%	33±10	103.1	20.8	140±10	103.0	23.9	180±10	102.8	23.7
20	20	Na ₂ SiO ₃ 2.0% + NH ₄ Cl 1.25%	450±5	101.7	22.8	270±10	100.4	25.2	320±10	100.5	24.5

¹ C. S. is actual compressive strength in psi

² D. D. is dry density at test, i. e., pounds dry solids per cubic foot

³ Per cent volatiles at test, i. e., weight lost upon drying as a percentage of dry solids weight

⁴ Basic magnesium carbonate (4 MgCO₃·Mg(OH)₂·4H₂O) used

⁵ Magnesium concentration equivalent to 1.25% MgSO₄·7H₂O

⁶ 2.50% NH₄Cl is equivalent to one quarter the stoichiometric amount required for combination with CaO
⁷ Sodium metasilicate (Na₂SiO₃·9H₂O) used, equivalent to 0.5N (1.30%) and 1.0N (2.0%) in molding water; samples molded at about 17% water content.

TABLE III-3b
EFFECT OF SECONDARY ADDITIVES ON THE STABILIZATION OF VBC WITH 5% CaO

Days Humid Cure Days Immersion	1			1			7		
	C. S. psi	D. D. pcf	Vols. %	C. S. psi	D. D. pcf	Vols. C. S. psi	D. D. pcf	Vols. %	
Control	270±5	104.9	17.0	125±5	104.5	21.4	150±5	103.4	22.3
+ MgSO ₄ ·7H ₂ O 1. 25%	360±10	106.2	16.7	235±15	103.6	20.6	270±5	105.6	20.9
+ ZnSO ₄ ·7H ₂ O 1. 46%	435±5	106.5	17.2	209±15	105.8	21.6	230±5	105.8	20.8
+ NiSO ₄ ·6H ₂ O 1. 34%	435±5	107.2	16.9	170±10	105.4	21.0	275±5	106.3	20.0
+ CuSO ₄ 0. 814%	350±5	107.0	15.9	170±15	106.6	20.9	180±10	106.5	20.4
+ Al ₂ (SO ₄) ₃ ·18H ₂ O 1. 69%	350±5	104.8	17.2	100±10	102.8	23.2	175±5	104.4	21.7
+ ZnSO ₄ ·7H ₂ O 1. 46% + Na ₂ SiO ₃ ·9H ₂ O 1. 54%	410±5	103.5	20.6	210±5	102.5	22.9	205±10	103.5	22.6
+ NiSO ₄ ·6H ₂ O 1. 34% + Na ₂ SiO ₃ ·9H ₂ O 1. 54%	395±5	104.5	19.8	190±5	103.4	22.6	280±10	104.1	21.8
+ CuSO ₄ 0. 814% + Na ₂ SiO ₃ ·9H ₂ O 1. 54%	350±5	103.0	20.8	180±10	101.5	23.6	200±15	102.8	22.8
+ MgSO ₄ ·7H ₂ O 1. 25% + Na ₂ SiO ₃ ·9H ₂ O 1. 3%	475±30	104.7	20.3	265±5	103.7	22.7	330±5	102.8	23.1

¹ Equivalent to 1. 25% MgSO₄·7H₂O

² Equiv. to 0.5N in molding water

TABLE III-4
STABILIZATION OF HBC WITH CaO AND SECONDARY ADDITIVES

Days Immersion	Days Humid Cure	1			1			1			7		
		C. S. psi	D. D. pcf	Vols. %									
+ CaO 5%	315±5	104.8	18.0	245±10	104.4	20.8	300±5	104.2	20.8	300±5	104.2	21.3	
+ CaO 5% (optimum)	290±5	102.0	20.5	260±5	102.0	21.8	410±5	101.0	21.8	410±5	101.0	22.4	
+ CaO 5%	315±5	104.5	19.5	250±25	104.5	21.6	355±5	104.0	21.6	355±5	104.0	21.2	
+ CaO 5%	185±5	97.0	24.3	170±5	95.5	26.1	265±5	96.5	26.1	265±5	96.5	24.8	
+ CaO 5%	290±5	102.2	21.2	235±15	102.4	22.6	325±5	103.2	22.6	325±5	103.2	21.5	
+ CaO 1%	230±5	106.2	20.8	80±5	104.1	22.2	100±5	104.8	22.2	100±5	104.8	21.9	
+ CaO 2%	300±5	105.0	21.0	315±25	105.2	21.0	525±25	105.0	21.0	525±25	105.0	21.0	
+ CaO 7%	185±5	101.2	19.2	100±5	100.8	23.2	185±5	101.2	23.2	185±5	101.2	23.2	
+ CaO 5% + MgSO ₄ ·7H ₂ O 1.25%	365±5	101.2	21.8	390±20	101.0	23.6	475±5	100.9	23.6	475±5	100.9	22.9	
+ CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.57% ¹	460±5	104.0	20.1	345±15	103.4	22.2	455±25	103.2	22.2	455±25	103.2	22.1	
+ CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.57% ¹	365±5	100.0	23.2	375±35	100.4	23.9	470±15	101.0	23.9	470±15	101.0	23.2	
+ CaO 5% + MgSO ₄ ·7H ₂ O 1.25% + Na ₂ SiO ₃ ·9H ₂ O 1.57% ¹	510±5	100.2	22.5	505±15	100.1	23.8	735±20	100.1	23.8	735±20	100.1	23.8	
+ CaO 5% + ZnSO ₄ ·7H ₂ O 1.46% ²	285±5	102.4	20.2	205±20	101.0	22.9	230±5	102.0	22.9	230±5	102.0	22.3	
+ CaO 5% + NiSO ₄ ·6H ₂ O 1.34% ²	460±5	104.0	19.8	450±5	103.5	22.4	495±10	103.9	22.4	495±10	103.9	22.2	

¹ Equiv. to 0.5N in molding water

² Equiv. to 1.25% MgSO₄·7H₂O

TABLE III-5
STABILIZATION OF SSS WITH CaO AND SECONDARY ADDITIVES

Days Immersion	Humid Cure	0			1			1			7		
		C. S. psi	D. D. pcf	Vols. %									
CaO 5%		245±5	110.2	20.1	30±5	95.5	30.0	50±5	100.5	100.5	25.4		
CaO 5%		170±5	103.5	23.7	30±5	90.0	34.0	65±5	96.0	96.0	28.1		
CaO 5%		210±5	107.2	21.2	65±5	98.4	26.6	30±5	93.5	93.5	31.8		
CaO 5%		290±5	113.5	17.4	30±5	105.3	27.9	40±5	107.0	107.0	24.9		
CaO 5% + MgSO ₄ ·7H ₂ O 1.25%		275±5	111.5	18.8	20±5	102.3	30.0	35±5	103.7	103.7	24.5		
CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.57% ¹		250±5	107.5	21.3	30±5	95.7	30.9	40±5	98.2	98.2	25.7		
CaO 5% + MgSO ₄ ·7H ₂ O 1.25% + Na ₂ SiO ₃ ·9H ₂ O 1.57% ¹													
CaO 5% + ZnSO ₄ ·7H ₂ O 1.46% ²		220±5	108.8	19.7	35±5	99.3	27.5	40±5	102.1	102.1	23.2		
CaO 5% + NiSO ₄ ·6H ₂ O 1.34% ²		210 ± 5	107.2	20.2	35±5	96.6	29.4	40±5	99.3	99.3	24.8		

¹ Equiv. to 0.5N in molding water

² Equiv. to 1.25% MgSO₄·7H₂O

TABLE III-6
SELECTED RESULTS OF VBC, HBC, SSS, AND M-21

Days Humid Cure Days Immersion	1			1			7			
	0	C. S. psi	D. D. pcf	Vols. %	C. S. psi	D. D. pcf	Vols. %	C. S. psi	D. D. pcf	Vols. %
VBC + CaO 5%		270±5	104.9	17.0	125±5	104.5	21.4	150±5	103.4	22.3
HBC + CaO 5%		290±5	102.0	20.5	260±5	102.0	21.8	410±5	101.0	22.4
SSS + CaO 5%		230±5	108.6	20.6	40±20	97.6	29.6	45±20	99.3	30.1
M-21 + CaO 5%					disintegrated					
VBC + CaO 5% + MgSO ₄ ·7H ₂ O 1.25%		360±10	106.2	16.7	235±15	103.6	20.6	270±5	105.6	20.9
HBC + CaO 5% + MgSO ₄ ·7H ₂ O 1.25%		365±5	101.2	21.8	390±20	101.0	23.6	455±25	103.2	22.1
SSS + CaO 5% + MgSO ₄ ·7H ₂ O 1.25%		275±5	111.5	18.8	20±5	102.3	30.0	35±5	103.7	24.5
M-21 + CaO 4% + MgSO ₄ ·7H ₂ O 1%		240	118.3	7.7	100	117.9	14.6	125±5	118.0	14.9
VBC + CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.3%		350±5	105.7	18.5	170±20	103.2	23.0	200±5	103.5	21.9
HBC + CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.57%		365±5	100.0	23.2	375±35	100.4	23.9	470±5	101.0	23.2
SSS + CaO 5% + Na ₂ SiO ₃ ·9H ₂ O 1.57%		250±5	107.5	21.3	30±5	95.7	30.9	40±5	98.2	25.7
VBC + CaO 5% + MgSO ₄ ·7H ₂ O 1.25% + Na ₂ SiO ₃ ·9H ₂ O 1.3%		450±5	101.7	22.8	270±10	100.4	25.2	320±10	100.5	24.5
HBC + CaO 5% + MgSO ₄ ·7H ₂ O 1.25% + Na ₂ SiO ₃ ·9H ₂ O		510±5	100.2	22.5	505±15	100.1	23.8	735±20	100.1	23.8
VBC + CaO 5% + ZnSO ₄ ·7H ₂ O 1.46% ²		435±5	106.5	17.2	200±15	105.8	21.6	230±5	105.8	20.8
HBC + CaO 5% + ZnSO ₄ ·7H ₂ O 1.46%		285±5	102.4	20.2	205±20	101.0	22.9	230±5	102.0	22.3
SSS + CaO 5% + ZnSO ₄ ·7H ₂ O 1.46%		220±5	108.8	19.7	35±5	99.3	27.5	40±5	102.1	23.2
VBC + CaO 5% + NiSO ₄ ·7H ₂ O 1.34% ²		435±5	107.2	17.9	170±10	105.4	21.0	275±5	106.3	20.0
HBC + CaO 5% + NiSO ₄ ·7H ₂ O 1.34%		460±5	104.0	19.8	450±5	103.5	22.4	495±10	103.9	22.2
SSS + CaO 5% + NiSO ₄ ·7H ₂ O 1.34%		210±5	107.2	20.2	35±5	96.6	29.4	40±5	99.3	24.8

¹ Equiv. to 0.5N

² Equiv. to 1.25% MgSO₄·7H₂O

³ Average strengths of 4 samples

IV. SOIL STABILIZATION WITH ASPHALT EMULSIONS

A. Introduction

Previous work on asphalt stabilization of soils has indicated that emulsions of cutback asphalt in water, stabilized with a fatty amine such as Duomeen T-Hydrochloride, have considerable promise as topically applied treatments for porous soils. The purpose of this study was to ascertain whether the method of application of a cationic asphalt emulsion influenced the strength, water permeability, and resistance to erosion of topically treated soil.

The effects of cutback solvent, emulsion dilution, and pretreatment of the soil with water on the strength, curing rate, and permeability to water have been studied on Ottawa sand treated with a Duomeen T-hydrochloride-stabilized asphalt emulsion.

B. Experimental Procedure

1. Emulsion Preparation

Emulsions with formulations similar to that shown in Table IV-1 were prepared with a Premier Dispersator. The cutback solvent was either unleaded gasoline from M. I. T. laboratory supplies or a 50-50 mixture of toluene and n-heptane. The emulsifier, Duomeen T, is a diamine derivative of tallow and was obtained from Armour and Company. The asphalt was 100/120 penetration from the Esso Everett Refinery. Tap water was used throughout and the HCl was reagent grade. The emulsions were all prepared via the inversion technique.

2. Preparation of Samples

The soil used was a natural Ottawa sand with a mean weight-average particle size of 180μ (sieve analysis) and a mean number average particle size of 120μ (microscopic count). The range of particle sizes was $20-350\mu$. The sand grains were slightly weathered, but still quite angular in shape.

Samples were prepared by placing sand in a ceramic Buechner funnel 5 inches in diameter or a 3.5-inch glass Buechner. The emulsion dosage was measured by weight and applied either as prepared, or diluted with water. Emulsions were applied either to dry soils or to soils that had been sprayed with enough water to thoroughly wet a 1/4-inch layer of soil immediately prior to the application of the emulsion (pre-wetting).

3. Testing of Stabilized Soils

The strengths of the stabilized soils were measured by penetration, forcing a flat brass piston, 1/4 inch in diameter, perpendicularly into a soil surface at a constant rate of 2.5 mm/ minute. The soil "penetration strength" was taken as the maximum stress developed in such a penetration. Each value of penetration strength reported is the average of several measurements on different areas of the same sample. For each sample, measurements were made one week after the application and at regular intervals thereafter until a constant value (termed the "ultimate penetration strength") was achieved.

The water permeability of stabilized soils was measured by maintaining a constant head of water (3/4 inches) above the soil and measuring the initial water flow rate (based on first two hours of the test) and the steady-state water flow rate. These tests were made on samples prepared in glass funnels so that any redistribution of asphalt during permeation could be observed. In some cases the amount of asphalt leached out of the treated layer was estimated by evaporating the total amount of water which passed through the sample during the permeability test to recover the solids. All of the permeability tests were conducted one week after the application of the asphalt.

The depth of penetration of the asphalt was determined by direct measurement after sectioning the treated samples. The percentage of voids was estimated as 39.3% from bulk density measurements and the density of quartz (Handbook of Chemistry, p. 226). From this information and the asphalt dosage the fraction of the voids filled with asphalt was calculated.

C. Results and Discussion

1. Soils Treated with Emulsions Prepared from Gasoline Cutbacks

Soils treated with emulsions prepared from gasoline cutbacks exhibited profuse foaming one day after application and this foaming often continued for several days. The resulting soils were entirely unsatisfactory, and it was found that the source of the foaming was the light ends dissolved in the gasoline. A 50-50 mixture of toluene and n-heptane did not produce foams, and this solvent was therefore used for the remainder of the work.

2. Soils Treated with Emulsions Prepared with a 50-50 Mixture of N-heptane and Toluene as Cutback Solvent

It was believed that a 50-50 mixture of n-heptane and toluene would simulate the solvent characteristics of gasoline, and an emulsion of the formulation shown in Table IV-1 appeared to have rheological behavior similar to the gasoline cutback emulsions. The results of a series of tests made with a single batch of this emulsion are shown in Tables IV-2 and IV-3.

It can be seen that all of the samples with an asphalt content in the treated portion of the soil corresponding to 28% or more of the soil void volume developed ultimate penetration strengths of approximately 50 psi. Variations from this value shown in Table IV-2 are believed to be within the experimental limits of reproducibility. Strength of unstabilized soil was virtually zero.

When undiluted emulsions were applied to dry sand, a dosage of 0.135 grams of asphalt per square centimeter of soil resulted in penetrations of 12-14 millimeters and asphalt contents corresponding to 47-56% of the soil voids. These samples showed penetration strengths of 10 to 17 psi after seven days cure, and required 24 days cure to develop the ultimate penetration strength.

Applying a 1/4 inch layer of water to the soil immediately prior to the application of undiluted emulsions increased the penetration to 23 to 25 mm for 0.135 gm asphalt/cm² dosage, and decreased the asphalt content to 28-30% of the void volume. The seven day penetration strength was increased to 18 to 23 psi and the time required to develop ultimate penetration strength was reduced to 14 days.

Sample number 10 was prepared with pre-wetted sand using an asphalt dosage of 0.067 g/cm². In addition, 30 cc. of water was added to the sample as emulsion uptake was complete. It was believed that such a procedure might lower the asphalt content of the treated layer by increasing the rate of emulsion penetration, but instead the depth of penetration decreased to 12 mm while the asphalt content remained at 28%.

Samples prepared from emulsions diluted with an equal volume of water developed seven-day penetration strengths of 20 psi and required 21 days to reach the ultimate penetration strength. The percentage of the voids filled could not be calculated since a large portion of the asphalt was unevenly deposited throughout the sample below the satisfactorily stabilized layer. Increasing the dilution to 2/1 gave essentially no stabilization.

Water Permeability

The water permeability of several samples is shown in Table IV-3. The initial permeability of a sample stabilized by applying an undiluted emulsion to dry sand was $1.5 \text{ ml/cm}^2\text{-hr}$. For a sample prepared with pre-wetted sand, however, the initial permeability was $32 \text{ ml/cm}^2\text{-hr}$ and the steady-state value was $21 \text{ ml/cm}^2\text{-hr}$. Diluting the emulsion with an equal volume of water resulted in rates comparable to pre-wetted samples, and 2/1 dilutions gave rates similar to untreated sand. The low asphalt content (30% of soil voids) of samples pretreated with water is likely responsible for their high permeabilities. Thus, though an asphalt content of 30% of the voids is adequate to produce the ultimate penetration strength, higher values (about 50% of the voids) are required to significantly lower the water permeability of the sand.

Further, Table IV-3 shows that during the permeability test 31% of the applied asphalt was leached out of the sample with 51% asphalt in the voids, while only 1 to 5% of the applied asphalt was leached out of the sample with 30% asphalt in the voids. It is felt that the mobility of the asphalt in the sample with a high asphalt content may be responsible for the ten-fold decrease in permeation which this sample showed before steady-state was achieved; and it was noted that during the test considerable amounts of the asphalt from the treated layer were deposited in the underlying sand, presumably in regions of high initial permeability.

D. Conclusions

1. When gasoline is used as a cutback solvent the soils produce foamed layers which result in low strength and high permeability to water.
2. Any topical treatment of Ottawa sand with an asphalt emulsion which deposits an amount of asphalt equal to or greater than 28% of the soil void volume results in a fully cured penetration strength of 50 psi but considerably greater asphalt contents (about 50% of soil voids) are required to markedly reduce the water permeability of the soil.
3. Low asphalt contents (about 30%) result when the soil is pretreated with water; the depth of penetration is proportional to the asphalt dosage.

E. Recommendations

The following additional studies are recommended:

1. A study of the water permeability of the soils as a function of time.
2. A study of the effect of diluting the emulsion with water prior to application to the soils in amounts less than one volume of water per volume of emulsion.
3. A study of the maximum asphalt dosage and penetration obtainable with Ottawa sand.
4. Because of the accompanying economic advantage, a study of the effect of lower concentrations of Duomeen-T is recommended.
5. The use of aluminum salts incorporated in the water phase of an emulsion with excess phosphoric acid may produce an aluminum phosphate gel in the soil voids and result in strengths greater than those produced by asphalt alone; it is suggested that the effect of these additives be investigated.

TABLE IV-1
EMULSION FORMULATION

	Wt. %	Approx. Vol. %
100/ 120 asphalt	33.5	
50/ 50 mixture of n-heptane and toluene ¹	16.6	50
Duomeen-T	2.0	
Tap water	46.0	50
HCl (≈37%)	2.0	

¹ 16.6% gasoline was used in initial studies.

TABLE IV-2
DEPTH OF PENETRATION & STRENGTH OF OTTAWA SAND
STABILIZED WITH DUOMEEEN T-HYDROCHLORIDE EMULSIONS¹
OF ASPHALT IN WATER

Sample No.	1	2	3	4	5	6	7	8	9	10
Sample Preparation	dry sand	dry sand	dry sand	pre-wet sand	pre-wet sand	pre-wet sand	dry sand	dry sand	dry sand	pre-wet
Time for Emulsion Penetration (seconds)	60	60	60	30	30	30	60	60	90	--
Vol. water / vol. emulsion	0 / 1	0 / 1	0 / 1	0 / 1	0 / 1	0 / 1	1 / 1	1 / 1	2 / 1	0 / 1
Asphalt dosage, g/cm ²	0.135	0.135	0.135	0.135	0.135	0.135	0.135	0.135	0.135	0.067
Thickness of dense asphalt layer, mm.	14	12	13	25	--	23	4 ²	25	1	12
Thickness of cured asphalt layer, 7 days cure, mm.	3-5	--	--	10	--	--	4	--	1	--
Per cent voids filled with asphalt in dense layer, %	47	56	51	28	--	30	--	--	--	28
7 days cure penetration strength, psi	10-17	--	--	18-25	--	--	20-23	--	3	--
Ultimate penetration strength, psi	50	54	52	50-58	44	50	54	34 ³	3	46
Time to develop ultimate strength, days	24	24	24	14	14	<22	21	30	--	17

¹ Emulsion formulation is presented in Table IV-2

² A 22 mm layer below the "dense" layer contained a non-uniform deposit of asphalt

³ This sample not representative because of an unsuccessful permeation test.

TABLE IV-3
**PERMEABILITY OF OTTAWA SAND STABILIZED WITH
DUOMEEN-T HYDROCHLORIDE EMULSIONS OF ASPHALT
IN WATER AFTER SEVEN DAYS CURE**

Sample No.	3	6	8	9
Sample preparation	dry sand	pre-wet sand	dry sand	dry sand
Vol. water / vol. emulsion	0/1	0/1	1/1	2/1
Asphalt dosage (g / cm ²)	0.135	0.135	0.135	0.135
Initial permeability (mil / cm ² -hr)	3.1	32	≈ 36 ²	--
Steady state permeability (ml / cm ² -hr)	0.25	21	--	approx. untreated sand
Percentage of asphalt leached out	31	1-5	--	95
Fraction voids filled with asphalt	51%	30%	--	--
Wet strength after permeation ³	19	13-16	--	--
Strength 1 day after permeation ³	27-34	27-32	--	--
Ultimate strength	52	50	34	3

¹ Emulsion formulation is presented in Table IV-1

² The surface of this sample was severely damaged during the initial permeability test

³ Pounds per square inch

V. SOIL STABILIZATION WITH AN IRON-ALUMINUM SALT
OF PHOSPHORIC ACID (TCB)

(S. B. Thesis by Hamid H. Bawany in Department of Chemical Engineering, 1961)

A. Introduction

One of the major problems of stabilizing heavy clays with acidic phosphorus compounds has been poor resistance to deterioration or disintegration on immersion in water. Ferric chloride and aluminum chloride have been found to be promising waterproofers for heavy clays stabilized by phosphoric acid, and it has been postulated that the ferric and aluminum ions reduce swelling of the montmorillonoid minerals in the soil by replacing the interlayer metallic cations (Reference 4).

The purpose of this investigation was to evaluate the effectiveness of a material known as TCB for stabilizing two soils, namely Massachusetts Clayey Silt (M-21) and Vicksburg Buckshot Clay (VBC). TCB is an iron-aluminum salt of phosphoric acid. It is coarsely crystalline, slightly hygroscopic, and slowly water soluble. The main components of this material are:

P ₂ O ₅	48.5%
Fe ₂ O ₃	13.4%
Al ₂ O ₃	4.12%
F	3.75%
CaO	3.65%

It has been reported () that 50% of the P₂O₅ in TCB is immediately soluble, while the rest is inactive.

Judging by its composition, TCB showed promise of being a good soil-stabilizer. P₂O₅ was recognized as the primary stabilizer; it would react with the water in the soil to form phosphoric acid which would then react with the alumina and aluminosilicate in the soil to form a cementitious material. It was expected that the Fe₂O₃ and Al₂O₃ would act as waterproofers, and that the fluorine would accelerate cure. Additional advantages of TCB were believed to be low cost, ease of handling, and the presence of all necessary primary and secondary stabilizing components in a single material.

B. Procedure

The properties of the soils investigated (M-21 and VBC) are presented in Table II-1.

The procedures used for mixing, compaction, and testing of samples were similar to those presented in Section II. Briefly, an amount of TCB corresponding to the desired treatment level (based on dry soil weight) was hand mixed with the soil. The mixture was then placed in a small finger-prong mechanical mixer and additional water was added slowly until the total liquids volume was 12.5 cc per 100 gm dry soil for M-21, and 17.5 cc per 100 gm dry soil for VBC. Previous work (1, 2) has indicated that maximum compacted densities are obtained at these liquid contents.

The samples were prepared by placing about 150 gm of the mixture in a Harvard miniature compaction mold (a cylinder, 2.76 in. by 1.36 in. in diameter) and subjecting it to two-end static compaction at about 1000 psi in a Carver hydraulic press.

In every batch seven samples were made, which were then cured for varying periods at room temperature over water in a sealed dessicator at 100% relative humidity. Four of them were cured for 24 hours; of these four samples, two were immediately tested after cure, and two were immersed in water for 24 hours and then blotted dry and tested. The remaining three samples of the batch were cured for seven days; of these three samples, two were tested immediately after cure, and the third was immersed in water for 24 hours and then blotted dry and tested. All samples were tested to failure in unconfined compression at a slow, constant rate of strain, to determine compressive strength. Also all samples prior to testing were weighed and their volumes determined by caliper measurements. After testing, samples were dried in an oven at 110°C for 24 hours and then weighed again. From these measurements dry density and volatile contents at testing were calculated.

Altogether six batches of seven samples each were made for Massachusetts Clayey Silt, at TBC treatment levels of 4, 5, 6, 7, 10, and 20 gms. per 100 gms. dry soil.

Three batches of seven samples each were prepared for VBC, at TBC treatment levels of 4, 10, and 20 gms. per 100 gms. dry soil.

C. Discussion of Results

1. Stabilization of M-21 with TCB (Table V-1)

Seven day humid cure strengths are on the average about 30% higher than 24 hour humid cure strengths, and immersed strengths are on the average about 65% lower than the corresponding humid cure strengths. Comparison of the results of this investigation with the results of previous studies involving phosphoric acid with and without added fluorine compounds indicates fluorine in TCB accelerates cure. The results of this investigation are also consistent with the proposed mechanism for phosphoric acid stabilization of the soil (Reference 1).

According to the proposed mechanism, phosphoric acid reacts with the alumina and aluminosilicates in the soil to form acid-soluble phosphates, followed by gelation as the acid is consumed. When the acid concentration is increased, the strength of the soil for short humid cure periods actually decreases. In a previous investigation (Reference 6) it was found that 24 hour humid cure strength for 5% phosphoric acid was lower than 24 hour humid cure strength for 2% phosphoric acid. The low 24 hour strength with 5% acid is believed to be due to a large amount of unreacted phosphoric acid solubilizing the aluminophosphate and preventing gelation. Addition of 0.5% sodium fluosilicate to 5% phosphoric acid was found (Reference 6) to greatly increase 24 hour humid cure strength, without affecting ultimate cured strength. It is believed that fluorides aid in removing a passivating film of absorbed phosphate which forms on the alumina, thereby accelerating the attack of alumina by the acid to form the gel.

The above considerations for phosphoric acid stabilization help to explain why the strength of M-21 increases with increasing concentrations of TCB. 10% TCB concentration (equivalent to 4% phosphoric acid) contains about 0.37% fluorine. It would, therefore, be expected that 24 hour humid cure strength at this concentration of TCB would lie somewhere between 2% phosphoric acid with 0.5% sodium fluosilicate and 5% phosphoric acid with 0.5% sodium fluosilicate. The results obtained indicate that this is the case. At 10% TCB, 24 hour humid cure strength is 318 psi, and this lies between 220 psi for 2% phosphoric acid with 0.5% sodium fluosilicate and 500 psi for 5% phosphoric acid with 0.5% sodium fluosilicate (Reference 6).

The results obtained with TCB at low concentrations are lower than those obtained with phosphoric acid stabilization. From Table V-1, at 5% TCB (equivalent to 2% phosphoric acid) the compressive strengths for 24 hour humid cure, 24 hour humid cure followed by 24 hour immersion, and 7 day humid cure are: 159, 29, and 166 psi, respectively.

The corresponding compressive strengths for 2% phosphoric acid alone are: 245, 44, and 305 psi. Previous investigation (Reference 1) has shown that strength varies with compacted density. For TCB stabilization the compacted densities are on the average about 7 pounds per cubic foot lower than phosphoric acid stabilization, and as a result compressive strengths are also lower. Moreover, it may be pointed out here that recent work on stabilizing M-21 with phosphoric acid (see Section II, this report) gave compressive strengths generally lower than earlier investigations (Reference 6). The reason for this may be due to variations in the soil.

At 20% TCB concentration (equivalent to 8% phosphoric acid) 24 hour humid cure strength is 447 psi. At this concentration, TCB contains about 74% fluorine. The strength of 447 psi is comparable to 500 psi with 8% phosphoric acid and 0.5% sodium fluosilicate obtained in previous investigation (Reference 6). One would expect higher strengths than those obtained with 20% TCB. It is believed that lower compacted densities and variations in the soil may be responsible for the slightly lower strengths.

From the results it is possible to determine the optimum TCB treatment level. The ratios of psi to per cent TCB at treatment levels of 4, 5, 6, 7, 10, and 20% TCB are (approximately): 27, 32, 42, 39, and 22 psi per per cent TCB, respectively. Thus, the optimum treatment level is that corresponding to 6% TCB concentration, and the 24 hour humid cure strength obtained at this level of treatment is 253 psi which is adequate for many applications.

Finally, it may be noted that comparability of results between TCB stabilization and the phosphoric acid stabilization confirms that P_2O_5 in TCB is the prime stabilizing agent as was expected.

2. Stabilization of VBC with TCB (Table V-2)

The results in Table V-2 show that 24 hour humid cure strengths of VBC stabilized with TCB increase with TCB concentration, as would be expected.

Seven day humidure strengths for the 4% and 10% TCB treatment levels were actually lower than the corresponding 24 hour humidure strengths. The reason for this probably is that at these levels of treatment, samples absorbed considerable water during humidure (see Table V-2). These facts suggest that water absorption may be responsible for the decreased strengths. Because of the extremely fine-grained nature of VBC, and because the water content during cure was significantly below optimum, the soil may well tend to absorb water during humidure at 100% relative humidity.

The samples with up to 10% TCB concentration disintegrated when immersed in water after both 24 hour humid cure and 7 day humid cure. For 20% TCB, appreciable immersed strength was obtained after 7 day humid cure, but the samples disintegrated when immersed in water after 24 hours humid cure. (Fe_2O_3) and aluminum oxide (Al_2O_3) in TCB did not give waterproofing action as had been expected. A further evidence of this can be seen by reference to Table V-2. The per cent volatiles at test after 7 day humid cure is 15.8 for 20% TCB concentration. The per cent volatiles after 7 day humid cure followed by 24 hour immersion is 22.4 for 20% TCB concentration. The high amount of water absorption on immersion shows the lack of waterproofing action by Fe_2O_3 and Al_2O_3 . The reason for this is not clear, but it might be that oxides reacted to form the phosphates instead of the ion-exchange reaction with the montmorillonoid minerals in VBC.

Overall considerations of VBC stabilization with TCB show that TCB alone is not very suitable as a stabilizer for Vicksburg Buckshot Clay.

3. Economic Aspects of TCB Soil Stabilization

TCB costs about \$60-90 per ton (Reference 7). If the figure of \$80 is assumed, then the cost of TCB equals 4 cents per pound.

To obtain strengths for M-21 comparable to those obtained with 2% phosphoric acid, 6% TCB on dry soil weight is required. Assuming an average soil density of 100 pounds per cubic foot (2700 pounds per cubic yard), 162 pounds of TCB are required to stabilize one cubic yard of the soil. Thus, the cost of stabilization would be \$6.48 per cu. yd. of the soil. The cost of stabilization with 2% phosphoric acid alone has been estimated (Reference 2) to be \$3.59 per cu. yd. soil. For stabilization phosphate rock-sulfuric acid mixture the cost is about \$.06 per cu. yd. soil.

The above cost figures indicate that TCB is not at present competitive with other stabilizing systems for Massachusetts Clayey Silt.

For Vicksburg Buckshot Clay very high concentration (20% or more TCB on dry soil weight) of TCB is needed, and since the price of TCB is quite high, stabilization of VBC with TCB is unsuitable.

D. Conclusions

1. TCB can successfully stabilize Massachusetts Clayey Silt. Although the compressive strengths obtained with TCB are somewhat lower than those obtained with phosphoric acid stabilization, they are adequate for stabilization purpose. The results of this investigation confirm that the P_2O_5 in TCB is the primary stabilizing agent.

2. The fluorine in TCB has a significant effect on cure acceleration as was expected.

3. TCB cannot successfully stabilize Vicksburg Buckshot Clay. Samples disintegrated on immersion in water at low concentrations of TCB.

4. Iron and aluminum oxides in TCB have little, if any, effect as waterproofers for VBC.

5. Economic considerations indicated that TCB is not at present competitive with other stabilizing systems for Massachusetts Clayey Silt, and entirely unsuitable for Vicksburg Buckshot Clay.

E. References

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4. Soil Stabilization by Chemical Methods, Final Report Phase XI, M.I.T., November 1959
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6. Soil Stabilization by Chemical Methods, Final Report Phase X, M.I.T., November 1958
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TABLE V-1
STABILIZATION OF M-21 WITH TCB
(Data average of 2 samples)

Days	Humid	Cure	1			1			7			7		
			0	0	0	1	1	1	0	0	0	C. S.	C. S.	C. S.
TCB % ²	C. S. psi ³	Vol. pcf ⁵	D. D. pcf ⁵	C. S. psi	Vol. %	D. D. pcf	C. S. psi	Vol. %	D. D. pcf	Vol. %	D. D. psi	C. S. Vol. psi	D. D. pcf	
H ₃ PO ₄														
4	1.6	107±8	10.7	124.8±0.3	32±8	12.8	124.1±0.7	167±8	10.1	124.1±0.1	48	12.6	123.3	
5	2	159±4	10.5	122.6±0.1	29±11	12.6	122.6±1.2	166±4	10.8	120.6±0.1	-	-	-	
6	2.4	253±9	10.3	124.8±0.1	63±9	12.7	123.2±0.1	315±65	10.0	122.8±1.1	78	13.9	121.4	
7	2.8	274±18	10.4	125.6±0.7	96±9	12.6	122.9±0.7	400±20	10.2	123.9±0.7	150	13.5	122.8	
10	4	318±24	11.6	125.9±0.1	175±7	12.8	125.1±0.3	420±20	11.4	123.4±0.6	230	13.3	123.3	
20		447±8	11.6	126.9±0.9	360	12.7	124.7±0.8	640±55	13.2	123.9±0.1	579	14.7	122.2	
2 ⁶		245±25	9.25	131	44±6	13.01	124	305±25	9	130.5	66	13.9	122	
5 ⁷		225±10	10.7	132.5	220	11.4	131.5							

¹ gms. TCB per 100 gms. dry soil

² Note that only 50% of the P₂O₅ in TCB is converted to H₃PO₄

³ Actual compressive strength in psi

⁴ % volatiles is weight lost upon drying as percentage of dry solids weight

⁵ Dry density is in pounds dry solids per cubic foot at test

⁶ From Reference 5

⁷ From Reference 6

TABLE V-2
STABILIZATION OF VBC WITH TCB
(Data average of 2 samples)

Days	Humid Cure	1	1	7	7
Days	Immersion	0	1	0	1
%					
TCB %	Equiv. H ₃ PO ₄ ²	C. S. psi ³	Vol. %	D. D. pcf ⁴	C. S. psi
4	1.6	130	14.6	104.4±0.6	Disintegrated
10	4	262±23	15.6	107.2±0.3	Disintegrated
20	8	320±5	15.0	108.8±0.8	Disintegrated
2 ⁶	240±5	16.9	112.0±0.2	Disintegrated	-
5 ⁶	385	-	-	Disintegrated	-

¹ gms. TCB per 100 gms. of dry soil

² Note that only 50% of the P₂O₅ in TCB is converted to H₃PO₄

³ Actual compressive strength in psi

⁴ % volatiles is weight lost upon drying as percentage of dry solids weight

⁵ Dry density is pounds dry solids per cubic foot at test

⁶ From Reference 2

VI. EVALUATION OF SOIL STABILIZERS

A. "Laboratory Comparison of Cement, Lime, and Phosphoric Acid Stabilization"--S. M. Thesis in Civil Engineering by G. Caro Mendoza, 1960

1. Scope

This work investigated the effects of compactive effort, type of compaction, time of humid cure, cycles of wetting and drying, and cycles of freezing and thawing on the soaked unconfined compressive strength of a stabilized clayey silt (Massachusetts Clayey Silt--M-21) and of a stabilized fat clay (Vicksburg Buckshot Clay--VBC).

The selection of additives and concentrations used for stabilization was based on results obtained during the past several years by the Soil Stabilization Laboratory of M. I. T. which gave the most economical stabilization for arbitrarily selected unconfined compressive strengths of 150 psi and 300 psi after 7 days humid cure and one day immersion. These strengths were selected as possible minimum values for sub-base and base course stabilization for secondary roads, respectively. The four soil-additive systems chosen were:

1. M-21 + 5% portland cement yielding 300 psi
2. M-21 + 1.5% phosphoric acid yielding 150 psi
3. VBC + 5% quicklime (CaO) + 1.2% magnesium sulfate* yielding 300 psi
4. VBC + 4% quicklime yielding 150 psi

2. Atterberg Limits for the Soil-Additive Systems

Tabulated below are the Atterberg limits for M-21 and VBC before and after treatment. The soil-additive systems were stored in sealed containers for one week prior to the determination of the Atterberg limits.

Soil-additive system	Liquid Limit	Plastic Limit	Plasticity Index
M-21	17.2%	11.5%	5.7%
M-21 + 5% cement	19.0%	16.2%	2.8%
M-21 + 1.5% H_3PO_4	19.0%	16.5%	2.5%

* Both 1.2% $MgSO_4$ and 1.2% $MgSO_4 \cdot 7H_2O$ were used, but only the results for the hydrated form are reported.

<u>Soil-additive system</u>	<u>Liquid Limit</u>	<u>Plastic Limit</u>	<u>Plasticity Index</u>
VBC	66, 0%	28, 3%	37, 7%
VBC + 4% quicklime	40, 5%	31, 5%	9, 0%
VBC + 5% quicklime + 1, 2% $MgSO_4 \cdot 7H_2O$	45, 0%	39, 5%	5, 5%

3. Effect of Compactive Effort

The soaked unconfined compressive strengths (7 days humid cure and 1 day immersion) were determined for the four soil-additive systems at three compactive efforts. Kneading compaction was obtained using the Harvard miniature apparatus with 25 blows per layer with a 40-lb. tamper. The compactive effort was altered by varying the numbers of layers per sample; 3, 5, and 10 layers were used.

Curves of dry density and strength versus molding moisture content at the three compactive efforts are plotted in Figures VI-1 through VI-4. The data are tabulated in Tables VI-1A through VI-1D.

The results show that:

- (1) Maximum strength was obtained at molding moisture contents at or slightly higher than the optimum for maximum dry density.
- (2) The optimum moisture content decreased slightly with increasing compactive effort.
- (3) There was a marked increase in strength with increase in compactive effort.
- (4) The strength depended mainly on dry density and not on molding moisture content.

4. Effect of Type of Compaction

To determine the effects of type of compaction on the strength of the four soil-additive systems, samples were statically compacted at the optimum moisture content and to the same dry density as obtained with the 10-layer kneading compaction.

A comparison of the strengths obtained from static and kneading compaction is given in Figure VI-5.

The results showed that at the same molding moisture content and dry density:

- (1) For the stabilized silty soil (M-21), samples statically compacted were stronger, after curing and soaking, than samples compacted by kneading compaction. The difference amounted to 100% when phosphoric acid was used, and 40% with 5% cement as the stabilizer.
- (2) For the stabilized clay (VBC), samples compacted by kneading compaction were stronger than those statically compacted, and the difference amounted to 20% using either 4% quicklime or 5% quicklime plus 1.2% magnesium sulfate as the stabilizer.

5. Effect on Curing Time

The soaked strengths of the four soil-additive systems, statically compacted*, were determined after one, seven, fourteen, and twenty-eight days humid cure at 100% relative humidity followed by one day complete immersion. The results are plotted in Figure VI-6 and tabulated in Tables VI-2A and VI-2B.

All the soil-additive systems gained strength with increasing time of humid cure. The gain in strength of M-21 plus 5% cement was much more pronounced during the 28-day period than the other stabilized systems.

6. Effect of Cycles of Wetting and Drying

Samples of the four soil-additive systems were statically compacted, humid cured for seven days, soaked for one day, and then subjected to 1, 2, 5 and 10 cycles of drying (15 hours at 75°C) and wetting (9 hours complete water immersion) after which their unconfined compressive strengths were determined.

* For the effects of time of curing, cycles of wetting and drying, and cycles of freezing and thawing, the samples were statically compacted at the optimum moisture content and to the same dry density as obtained for the 10-layer kneading compaction.

The results for M-21 plus 5% cement and M-21 plus 1.5% H_3PO_4 are plotted in Figure VI-7. The samples for VBC stabilized with 4% quicklime and 5% quicklime plus 1.2% magnesium sulfate disintegrated after one cycle of drying and wetting. The data on the two soils are presented in Tables VI-3A and VI-3B.

The conclusions drawn from these tests were:

- (1) Wetting and drying action was beneficial for M-21, especially when stabilized with 5% cement.
- (2) One to two cycles of drying and wetting were sufficient to cause complete disintegration of the VBC samples stabilized with 4% quicklime or 5% quicklime plus 1.2% $MgSO_4 \cdot 7H_2O$.

7. Effect of Cycles of Freezing and Thawing

For the freeze-thaw tests the samples were compacted and cured in the same manner as for the wet-dry test samples, after which they were subjected to 1, 2, 5, and 10 cycles of freezing (15 hours at $14^{\circ}F$ during which time the samples had access to water) and thawing (9 hours at $84^{\circ}F$ and ~95% relative humidity). The samples were then soaked for one day prior to testing.

The results, plotted in Figures VI-8 and VI-9 and tabulated in Tables VI-4A and VI-4B, showed that:

- (1) Only M-21 plus 5% cement withstood 10 cycles of freezing and thawing.
- (2) VBC plus 5% CaO plus 1.2% $MgSO_4 \cdot 7H_2O$ withstood five cycles of freezing and thawing.
- (3) Samples of M-21 stabilized with 1.5% H_3PO_4 and samples of VBC with 4% CaO were completely disintegrated after five cycles of freezing and thawing.

8. Conclusions

For the soil-stabilizer systems investigated the following general conclusions may be drawn:

- (1) Maximum soaked strengths were obtained at water contents at or slightly higher than the water contents for maximum

dry density for any given compactive effort. For all practical purposes maximum strength may be considered to occur at the water content for maximum dry density.

- (2) An increase in compactive effort (i.e., an increase in dry density) caused an increase in strength at any given water content.
- (3) The strain at failure increased with increase in water content. At any given water content, an increase in compactive effort caused an increase in strain at failure.
- (4) The longer the time of humid cure the higher the compressive strength after immersion.

The results of the freeze-thaw and wet-dry tests on stabilized M-21 suggest that a minimum soaked strength of 300 psi is reasonable for base course construction but that the strength value of 150 psi for subbase construction may be too low in climates conducive to extreme frost action. The results of the cyclic tests on the fat clay, VBC, clearly show that strengths of 300 and 150 psi are too low to withstand the detrimental effects of wetting and drying. However, such strengths are only slightly unconservative under severe conditions of freezing and thawing. It is concluded therefore that in the use of soaked unconfined compressive strengths as design criteria for stabilized soils the strength values selected should be varied according to the type of soil and the type of climate.

B. "Comparative Cost of Stabilizers"--S. B. Thesis in Civil Engineering, B. T. K. Adadevoh, 1960

The comparative cost of stabilization of three soils was considered, namely, New Hampshire Silt, Massachusetts Clayey Silt, and Vicksburg Buckshot Clay. The stabilizer formulations were obtained from M. I. T. Soil Stabilization reports. Interpolation was used to obtain formulations which would yield unconfined compressive strengths of 150 and 300 psi after 7 and 28 days humid cure (at 100% relative humidity) followed by one day complete immersion. The strength values were selected as possible minimum values for subbase and base course stabilization for secondary roads respectively.

The prices of the chemicals reported are listed in Table VI-5. A partial tabulation of the results for the most commonly used and better formulations are given in Tables VI-6, VI-7, and VI-8 for the three soils. It is shown that:

- (1) Quicklime was the most economical stabilizer for Vicksburg Buckshot Clay at a cost of \$0.40 per cu. yd. of dry soil for a soaked strength of 150 psi. For a strength of 300 psi, quicklime plus magnesium sulfate was the least expensive stabilizer at \$1.57 per cu. yd.
- (2) With Massachusetts Clayey Silt (M-21) portland cement Type I was the least expensive stabilizer at a cost of \$1.07 per cu. yd. for a seven-day strength of 150 psi or a 28-day strength of 300 psi. The cost for 7-day strength of 300 psi was \$2.20 per cu. yd. of dry soil.
- (3) The cheapest stabilizer for New Hampshire silt was portland cement plus sodium sulfate giving strengths after 7 days humid cure of 150 and 300 psi at a cost of \$1.95 and \$2.60 respectively. The cost for a strength of 300 psi after 28 days cure was \$2.07.

It should be pointed out that the data available for these comparisons did not always allow selection of the most economical formulations. For example, in many cases tests were run in which the per cent primary additive (e.g., cement) was kept constant and only the per cent secondary additive was varied. Also, the selection of the strength values was quite arbitrary and should really have been varied from soil to soil, and with the different types of stabilization. For example, an asphalt-stabilized soil would not require as high an unconfined strength as a cement stabilized soil for satisfactory performance. Also, as shown in the previous section of this report, VBC requires higher strengths for satisfactory performance than M-21 when subjected to cycles of wetting and drying.

TABLE VI-1A
**EFFECT OF COMPACTIVE EFFORT ON THE DRY DENSITY
 AND WET COMPRESSIVE STRENGTH OF M-21
 PLUS 5% CEMENT**

Points	At Molding		After Immersion		U.C.S. *** psi
	W*	D**	W	D	
<u>10 Layers</u>					
1	6.8	119.2	13.3	118.1	64
2	8.3	124.4	12.1	123.5	138
3	9.1	127.8	12.0	127.5	158
4	10.1	129.0	11.0	120.0	231
5	10.9	127.1	12.0	126.1	116
6	12.2	124.1	12.7	123.8	112
<u>5 Layers</u>					
1	8.0	120.0	12.0	119.5	95
2	10.8	126.9	11.6	126.0	165
3	11.1	126.2	12.5	124.7	150
<u>3 Layers</u>					
1	7.2	116.5	14.6	115.5	52
2	10.4	125.2	11.9	124.8	150
3	12.6	122.1	12.8	122.0	81

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After seven days cure and one day immersion

TABLE VI-1B

**EFFECT OF COMPACTIVE EFFORT ON DRY DENSITY
AND WET COMPRESSIVE STRENGTH OF M-21
PLUS 1.5% PHOSPHORIC ACID**

Points	At Molding		After Immersion		U.C.S. *** psi
	W *	D **	W	D	
<u>10 Layers</u>					
1	8.0	128.5	10.0	128.3	76
2	9.0	131.8	9.9	131.2	92
3	9.5	131.8	10.6	131.4	96
4	9.8	131.5	10.2	131.4	98
5	11.0	128.2	11.3	128.0	82
6	12.0	126.2	11.6	126.0	76
<u>5 Layers</u>					
1	7.3	118.4	13.6	118.1	58
2	9.6	128.1	11.5	127.5	66
3	10.0	129.2	10.6	128.9	72
4	12.2	124.1	12.8	123.8	42
<u>3 Layers</u>					
1	7.6	114.9	13.1	114.8	24
2	9.6	124.0	13.1	121.5	45
3	12.3	122.1	13.1	122.0	33

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After seven days cure and one day immersion

TABLE VI-1C

**EFFECT OF COMPACTIVE EFFORT ON DRY DENSITY
AND WET COMPRESSIVE STRENGTH OF VBC
PLUS 4% QUICKLIME**

Points	At Molding		After Immersion		U. C. S. *** psi
	W*	D**	W	D	
<u>10 Layers</u>					
1	16.6	98.7	23.6	97.5	68
2	17.2	98.3	23.7	98.1	84
3	17.7	99.4	23.2	99.0	82
4	21.5	103.0	23.6	100.5	189
5	23.7	100.5	24.8	98.8	166
6	24.2	99.8	24.4	98.3	151
<u>5 Layers</u>					
1	21.1	97.0	23.1	96.7	107
2	25.4	97.1	25.7	97.0	126
3	31.5	88.5	30.2	89.5	63
<u>3 Layers</u>					
1	20.8	95.2	22.8	95.0	77
2	24.1	96.1	24.7	95.0	86

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After seven days cure and one day soak

TABLE VI-1D

**EFFECT OF COMPACTIVE EFFORT ON DRY DENSITY
AND WET COMPRESSIVE STRENGTH OF VBC
PLUS 5% QUICKLIME PLUS 1.2% $MgSO_4 \cdot 7H_2O$**

Points	At Molding		After Immersion		U.C.S. *** psi
	W*	D**	W	D	
<u>10 Layers</u>					
1	14.4	97.7	27.0	96.7	56
2	18.2	99.2	23.6	99.0	153
3	19.2	100.0	23.3	100.0	150
4	21.8	100.2	22.8	100.0	242
5	23.0	99.7	23.9	99.5	214
<u>5 Layers</u>					
1	17.4	94.0	25.4	93.5	69
2	18.3	95.2	26.5	95.0	90
3	24.2	96.0	26.3	95.5	136
4	24.8	96.7	26.8	94.0	130
<u>3 Layers</u>					
1	18.3	92.0	27.6	91.5	57
2	21.4	97.5	25.1	95.4	97
3	26.2	93.2	28.7	91.4	50

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After seven days cure and one day soak

TABLE VI-2A
 PROPERTIES OF M-21 STABILIZED SOIL
 AT VARIOUS CURING AGES

Additive	At Molding		Curing Time days	After Immersion		U.C.S. *** psi
	W*	D**		W	D	
1.5% H_3PO_4	9.8	132.0	1	10.0	132.1	125±25
1.5% H_3PO_4	9.8	132.0	7	10.7	131.2	100±8
1.5% H_3PO_4	9.8	132.1	14	10.3	131.8	190±20
1.5% H_3PO_4	9.8	131.7	28	10.3	130.8	208±10
5% Cement	10.4	130.1	1	10.8	129.0	225±25
5% Cement	10.4	129.8	7	11.3	129.6	328±10
5% Cement	10.0	129.9	14	10.6	129.5	416±10
5% Cement	10.0	130.1	28	10.9	130.0	536±15

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After one day immersion

TABLE VI-2B
 PROPERTIES OF V. B. C. STABILIZED SOIL
 AT VARIOUS CURING AGES

Additives	At Molding		Curing Time days	After Immersion		U.C.S.*** psi
	W*	D**		W	D	
4% CaO	20.5	102.8	1	22.7	102.1	116±5
4% CaO	21.0	103.1	7	22.0	102.8	154±25
4% CaO	21.0	103.0	14	21.3	101.5	170± 8
4% CaO	21.0	102.9	28	22.9	101.8	239±10
5% CaO plus 1.2% MgSO ₄ ·7H ₂ O	20.7	100.6	1	28.0	100.0	120± 7
5% CaO plus 1.2% MgSO ₄ ·7H ₂ O	21.0	100.5	7	28.5	100.0	198±20
5% CaO plus 1.2% MgSO ₄ ·7H ₂ O	21.1	100.2	14	28.5	99.8	244±14
5% CaO plus 1.2% MgSO ₄ ·7H ₂ O	21.0	100.4	28	27.7	99.9	278±10
5% CaO plus 1.2% MgSO ₄	20.8	100.2	1	25.7	99.9	42± 4
5% CaO plus 1.2% MgSO ₄	21.0	100.3	7	24.7	100.0	135±20

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After one day immersion

TABLE VI-3A
 EFFECT OF WETTING AND DRYING
 CYCLES ON M-21 STABILIZED SOIL

Additives	No.					
	At Moldling	of	At Testing	U.C.S. ***		
	W*	D**	Cycles	W	D	psi
1.5% H_3PO_4	10.0	131.9	1	10.7	131.0	184±10
1.5% H_3PO_4	10.0	131.5	2	10.8	130.0	215± 2
1.5% H_3PO_4	10.0	131.5	5	10.6	130.3	223± 0
1.5% H_3PO_4	10.0	131.5	10	10.8	129.9	236±15
5% Cement	10.0	129.6	1	11.0	129.0	542±15
5% Cement	10.0	129.5	2	10.3	129.0	547±25
5% Cement	10.0	129.8	5	11.6	129.0	578±30
5% Cement	10.0	129.8	10	11.5	129.7	629± 8

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After 24 hours immersion

TABLE VI-3B
 EFFECT OF WETTING AND DRYING
 CYCLES ON VBC STABILIZED SOIL

Additives	At Molding		No. of Cycles	At Testing		U.C.S.*** psi
	W*	D**		W	D	
4% CaO	21.0	102.5	1	Disintegrated		
4% CaO	21.0	102.4	2	Disintegrated		
4% CaO	21.0	102.7	5	Disintegrated		
4% CaO	21.0	102.8	10	Disintegrated		
5% CaO plus						
1.2% MgSO ₄ ·7H ₂ O	21.0	100.0	1	26.1	99.7	154±10
5% CaO plus						
1.2% MgSO ₄ ·7H ₂ O	21.0	100.3	2	Disintegrated		
5% CaO plus						
1.2% MgSO ₄ ·7H ₂ O	21.0	100.0	5	Disintegrated		
5% CaO plus						
1.2% MgSO ₄ ·7H ₂ O	21.0	100.2	10	Disintegrated		

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After 24 hours immersion

TABLE VI-4A
EFFECT OF FREEZING AND THAWING
CYCLES ON M-21 STABILIZED SOIL

Additives	No.						U.C.S. *** psi
	At Molding W*	of D**	Cycles	At Testing W	D		
1.5% H ₃ PO ₄	9.7	132.0	1	12.2	130.5		26± 0
1.5% H ₃ PO ₄	9.7	132.1	2	11.4	131.5		54± 5
1.5% H ₃ PO ₄	9.7	131.9	5	Disintegrated			
1.5% H ₃ PO ₄	9.7	132.0	10	Disintegrated			
5% Cement	9.8	130.5	1	11.8	129.9		233± 2
5% Cement	9.8	130.2	2	11.6	128.8		266± 1
5% Cement	9.8	130.5	5	11.6	129.8		538± 6
5% Cement	9.8	130.5	10	11.8	129.2		456±20

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After 24 hours immersion

TABLE VI-4B
 EFFECT OF FREEZING AND THAWING
 CYCLES ON VBC STABILIZED SOIL

Additives	No.					U.C.S. *** psi
	At Molding W*	of D**	Cycles	At Testing W	D	
4% CaO	21.0	103.5	1	25.6	101.5	122±5
4% CaO	21.0	103.4	2	29.0	99.0	126±10
4% CaO	21.0	103.2	5	Disintegrated		
4% CaO	21.0	103.5	10	Disintegrated		
5% CaO plus						
1.2% MgSO ₄ . 7H ₂ O	21.0	103.0	1	28.6	99.0	118±20
5% CaO plus						
1.2% MgSO ₄ . 7H ₂ O	21.0	102.5	2	28.3	99.5	188± 0
5% CaO plus						
1.2% MgSO ₄ . 7H ₂ O	21.0	102.9	5	28.0	99.7	115 (1)
5% CaO plus						
1.2% MgSO ₄ . 7H ₂ O	21.0	103.0	10	Disintegrated		

* Moisture content in % of dry solids

** Dry density in pounds per cubic foot

*** After 24 hours immersion

(1) Result of one sample only

TABLE VI-5
PRICES OF CHEMICALS*

Name	Description	Price, dollars / lb.
Hydrated Lime		0.0088
Quicklime		0.0071
Magnesium Carbonate		0.14
Magnesium Sulfate		0.0235
Portland Cement	Type I	0.0143
Sodium Aluminate		0.085
Sodium Carbonate	Monohydrated	0.031
Sodium Hydroxide		0.041
Sodium Metasilicate	Anhydrous	0.057
Sodium Silicate		0.0155
Sodium Sulfate	Anhydrous	0.028

* Prices were obtained from several sources. The cost of many of the chemicals varies considerably with grade and amount purchased and thus the prices quoted above are only representative.

TABLE VI-4
PARTIAL SUMMARY OF THE COST OF STABILIZERS
FOR VICKSBURG BUCKSHOT CLAY

Stabilizer Formulation		Humid Cure (Days)	Soaked Unconfined Compressive Strength psi	Cost (\$/ cu.yd. of Soil)
Additives	Wt. / 100 lb. of Dry Soil			
Quicklime	2.0	7	150	0.40
Quicklime Magnesium Sulfate	2.6 0.65	7	150	0.91
Hydrated Lime Sodium Hydroxide	5.0 0.10	7	150	1.34
Hydrated Lime Sodium Sulfate	5.0 0.10	7	150	1.11
Hydrated Lime Sodium Metasilicate	5.0 0.25	7	150	1.11
Quicklime Magnesium Sulfate	4.64 1.16	7	300	1.57

TABLE VI-7
PARTIAL SUMMARY OF THE COST OF STABILIZERS
FOR MASSACHUSETTS CLAYEY SILT

Additives	Stabilizer Formulation		Humid Cure (Days)	Soaked Unconfined Compressive Strength psi	Cost (\$/ cu. yd. of Soil)
	Wt. / 100 lb. of Dry Soil				
Portland Cement*	2.35		7	150	1.07
Hydrated Lime	5.0				
Sodium Sulfate	0.50		7	150	1.77
Hydrated Lime	5.0				
Sodium Metasilicate	1.60		7	150	4.07
Portland Cement	5.0		7	300	2.20
Hydrated Lime	5.0				
Sodium Sulfate	1.60		7	300	2.68
Hydrated Lime	5.0				
Sodium Metasilicate	2.40		7	300	5.45
Portland Cement	< 2		28	150	< 1.00
Hydrated Lime	5.0				
Sodium Sulfate	0.50		28	150	1.77
Hydrated Lime	5.0				
Sodium Metasilicate	0.75		28	150	2.66
Portland Cement	2.35		28	300	1.07
Hydrated Lime	5.0				
Sodium Sulfate	0.75		28	300	1.97
Hydrated Lime	5.0				
Sodium Metasilicate	1.50		28	300	3.92

* Type I Portland Cement

TABLE VI
PARTIAL SUMMARY OF THE COST OF STABILIZERS
FOR NEW HAMPSHIRE SILT

Additives	Stabilizer Formulation	Wt. / 100 lb. of Dry Soil	Soaked Unconfined Compressive Strength psi			Cost (\$/ cu. yd. of Soil)
			Humid	Cure (Days)		
Cement*		5.0				
Sodium Sulfate		0.20	7		150	1.95
Cement		5.0				
Sodium Carbonate		0.25	7		150	2.03
Cement		5.0				
Sodium Aluminate		0.15	7		150	2.15
Cement		6.2	7		150	2.22
Cement		5.0				
Sodium Metasilicate		0.30	7		150	2.24
Cement		5.0				
Sodium Sulfate		1.15	7		300	2.60
Cement		5.0				
Sodium Aluminate		0.70	7		300	3.32
Cement		10.2	7		300	3.55
Cement		10.0				
Sodium Sulfate		0.25	7		300	3.64
Cement		5.0				
Sodium Sulfate		0.35	28		300	2.07
Cement		5.0				
Sodium Aluminate		0.25	28		300	2.35
Cement		5.0				
Sodium Metasilicate		0.55	28		300	2.60

*Type I Portland Cement

Figure VI-1

COMPACTION STRENGTH CURVE
M-21-plus 5% Cement

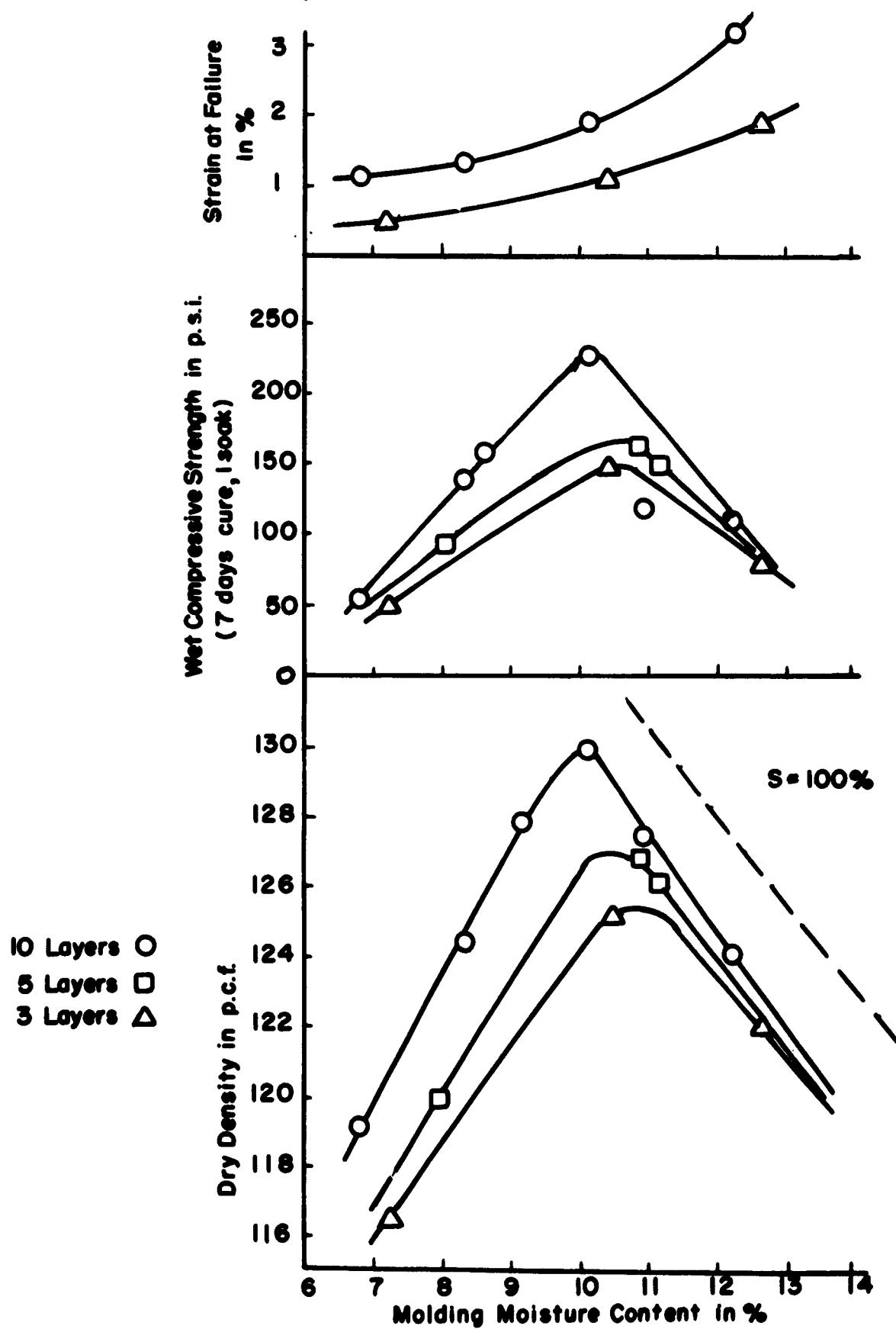


Figure VI-2
COMPACTION STRENGTH CURVE
M-21 plus 1.5% H_3PO_4

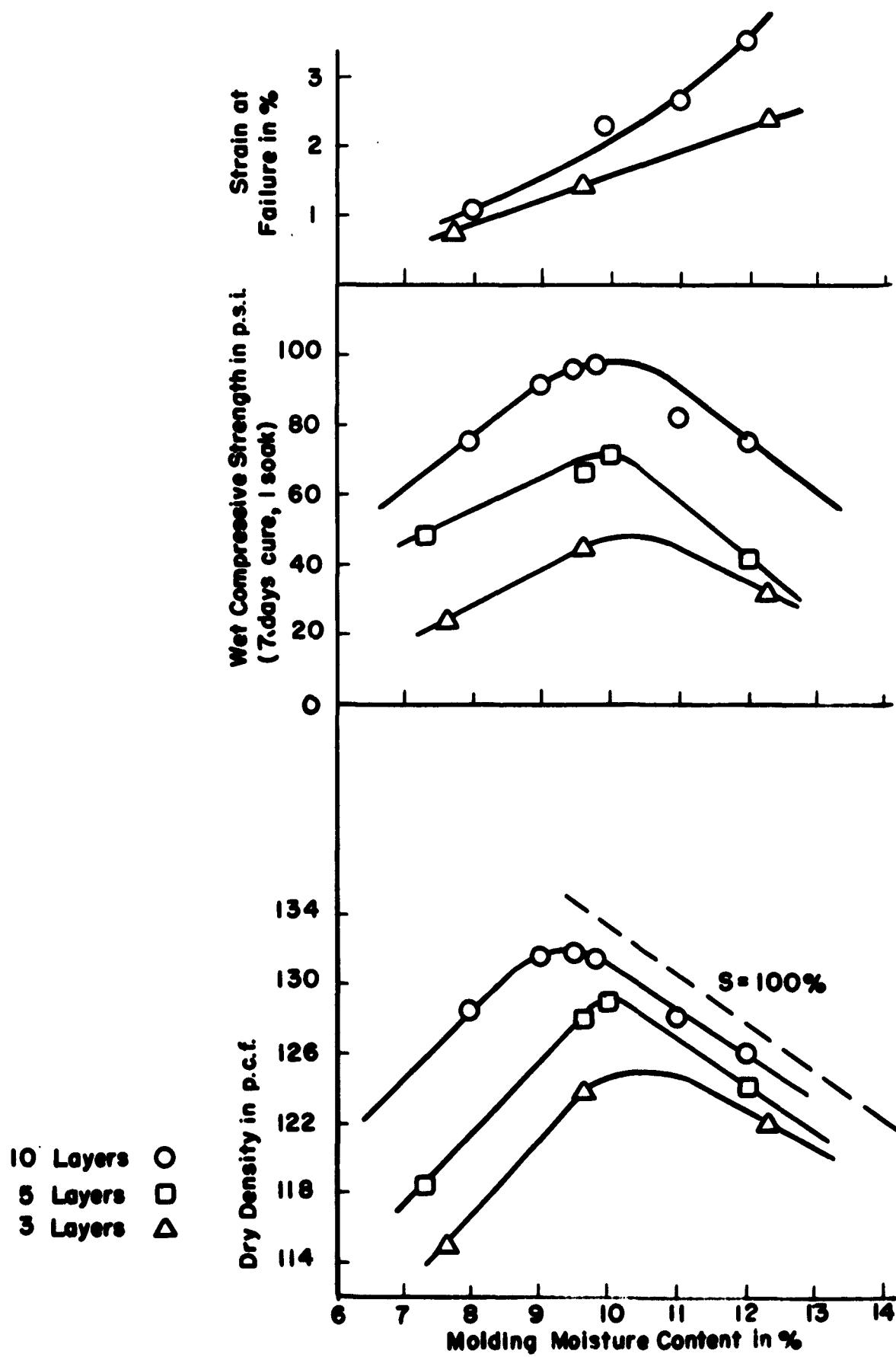


Figure VI-3

COMPACTION STRENGTH CURVE

V. B. C. plus 4% CaO

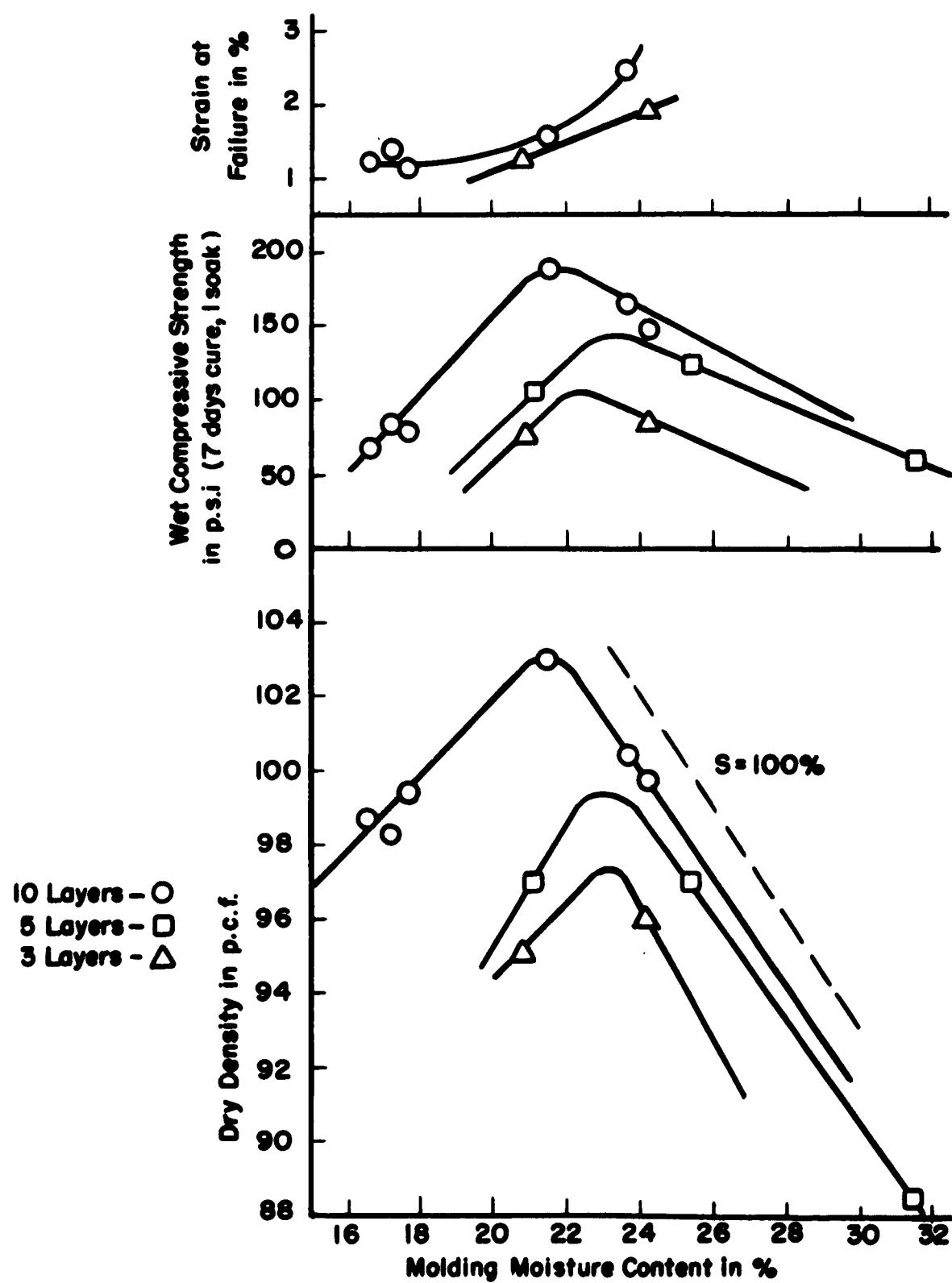


Figure VI-4

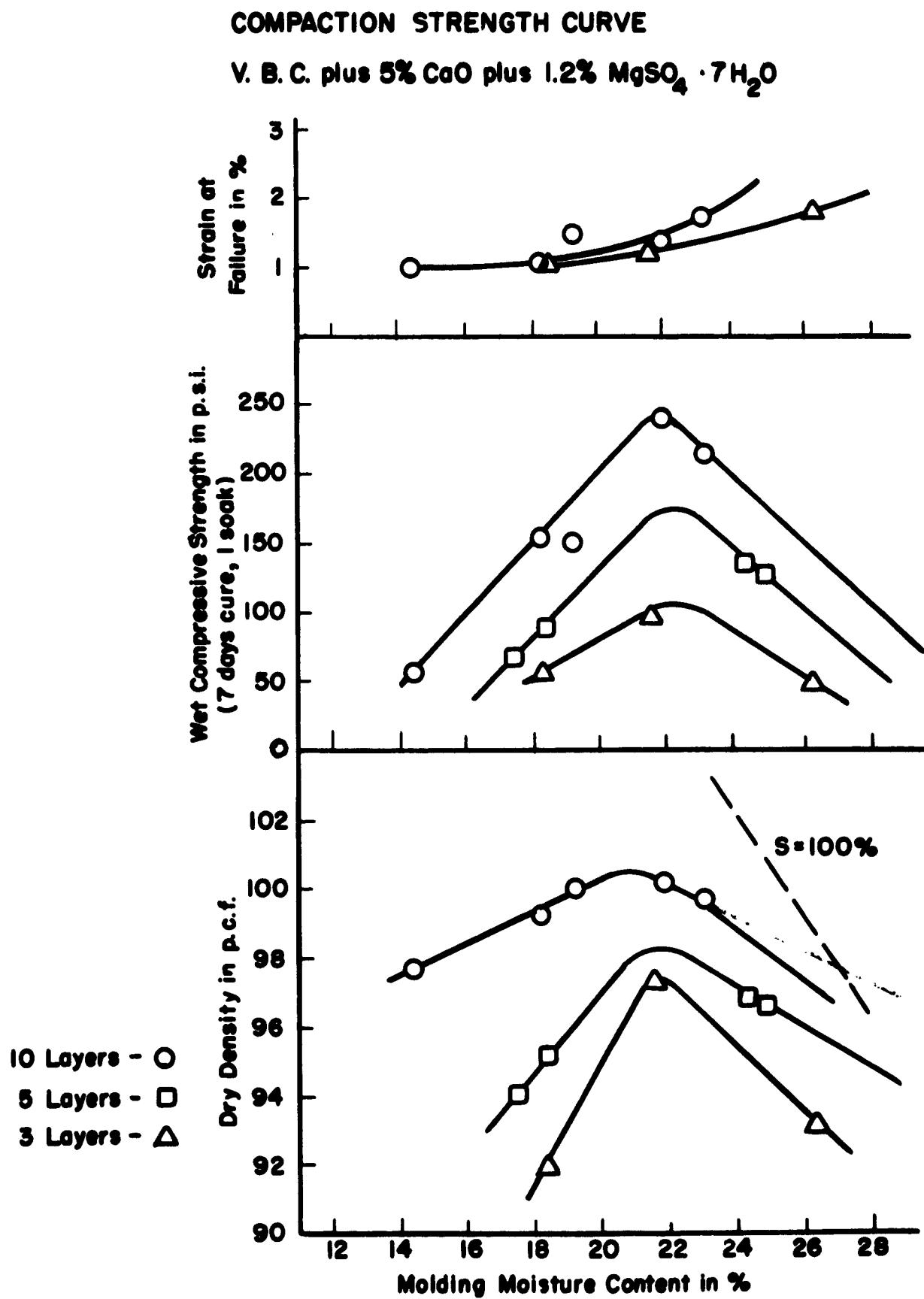
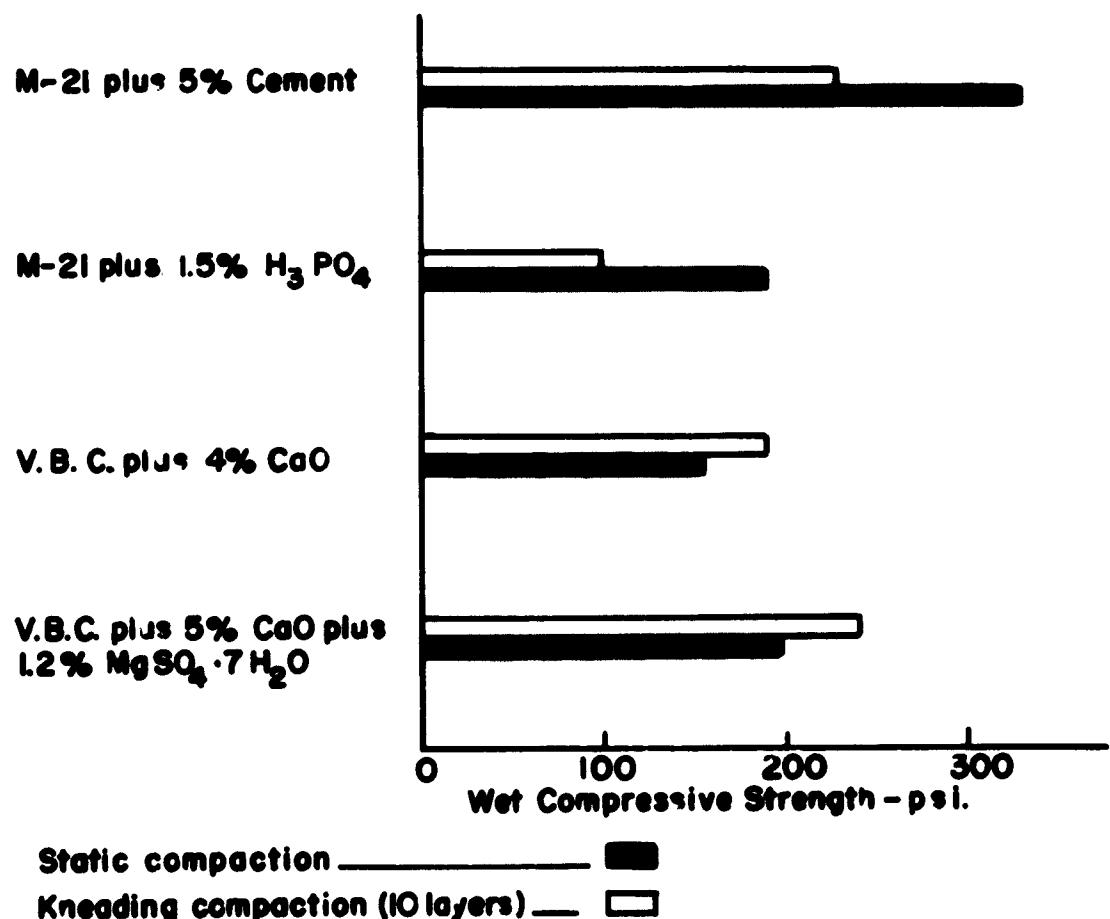


Figure VI-5

EFFECT OF TYPE OF COMPACTION
ON WET COMPRESSIVE STRENGTH
(AFTER 7 DAYS CURE AND 1 DAY SOAK)

Static D* = Kneading - 10 layers - $D \pm \frac{1}{2}$ pcf.



*Dry density in pcf.

EFFECT OF CURING TIME, STRENGTH OF SOIL-ADDITIVE SYSTEMS
ON

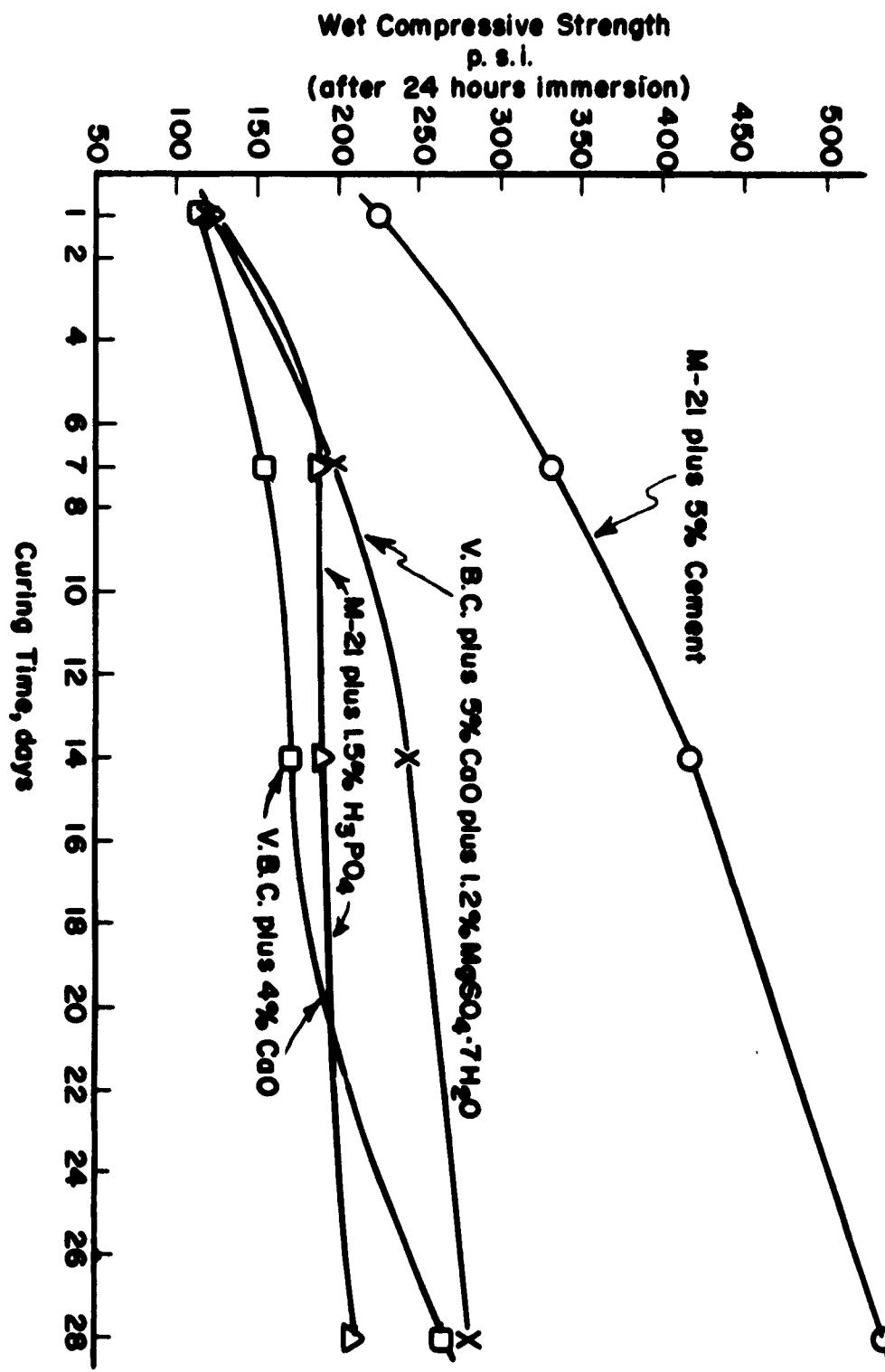


Figure VI-6

EFFECT OF WETTING AND DRYING CYCLES ON M-2I STABILIZED SOIL

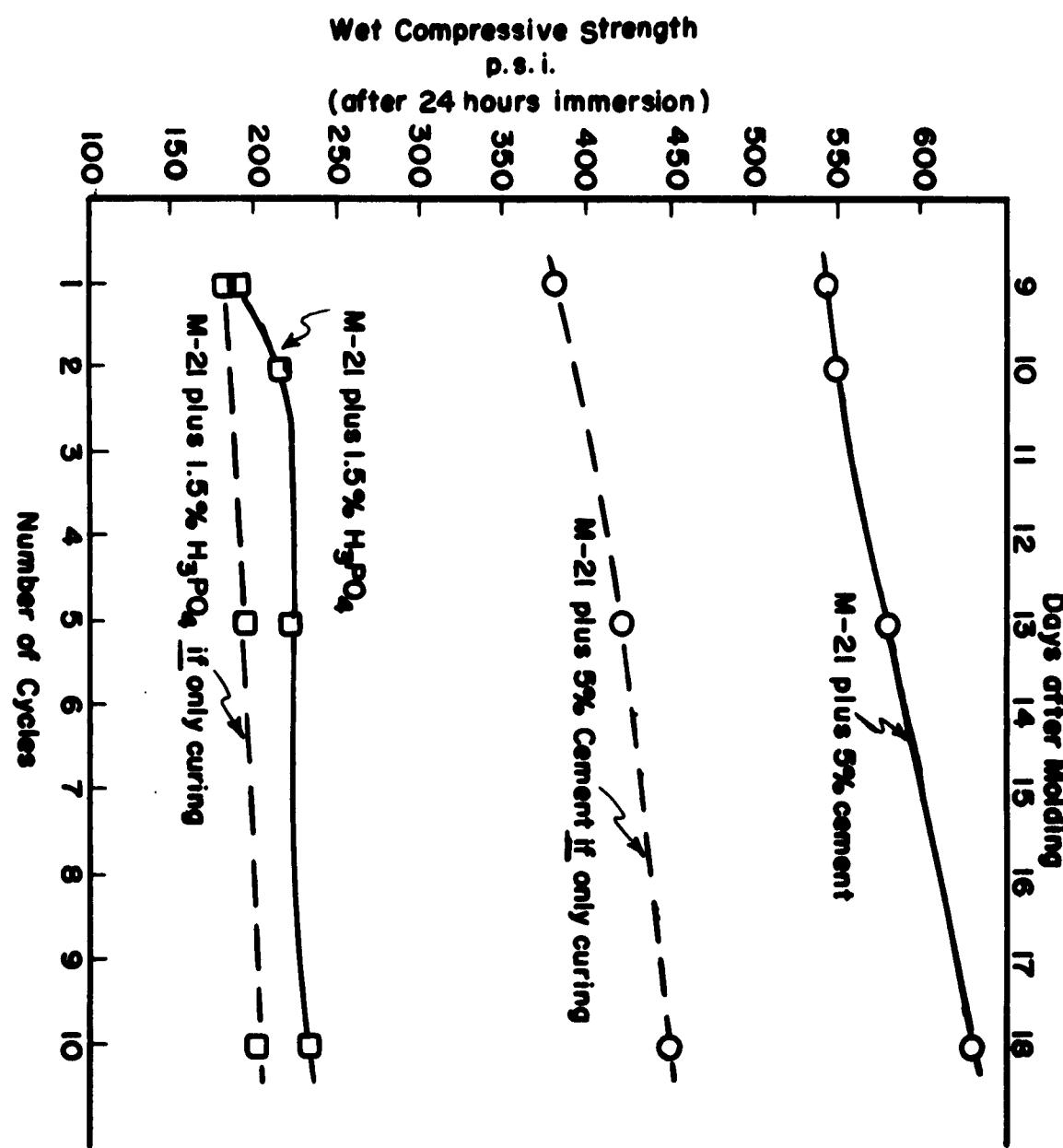


Figure VI-7

EFFECT OF FREEZING AND THAWING CYCLES ON M-2I STABILIZED SOIL

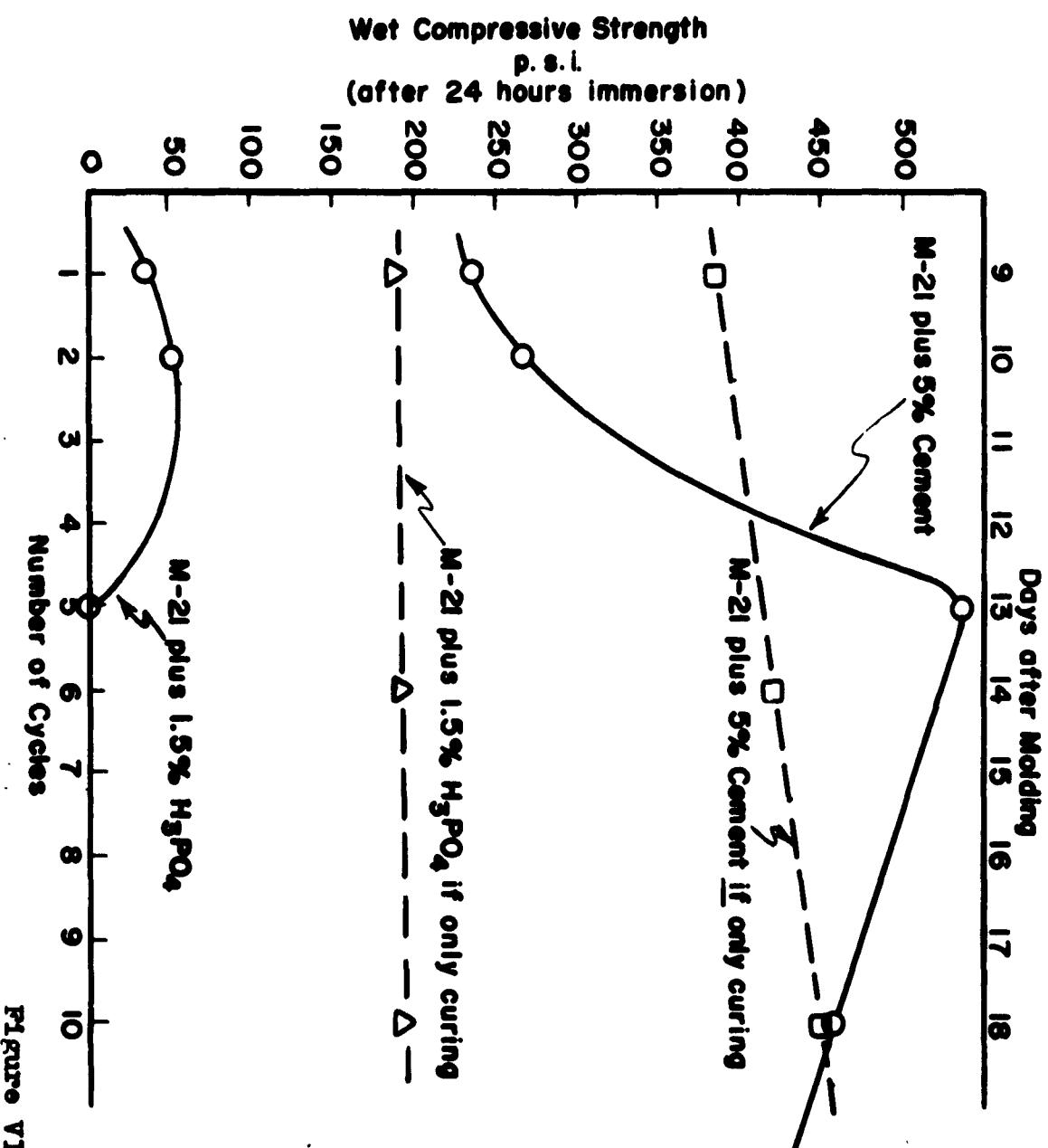


Figure VI-8

EFFECT OF FREEZING AND THAWING CYCLES ON V.B.C. STABILIZED SOIL

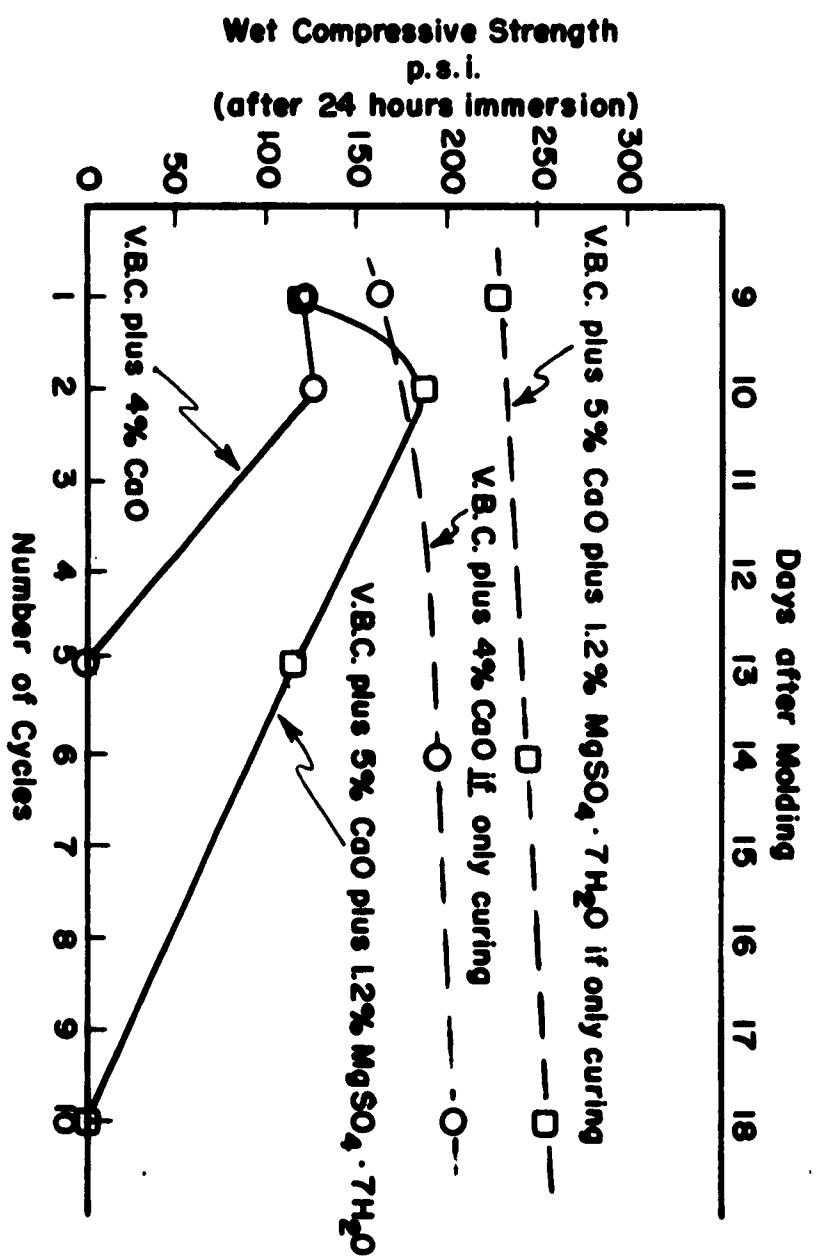


Figure VI-9

VII. EFFECTIVE STRESSES IN STABILIZED SOILS

A. Introduction

One of the most valuable and powerful tools that the soil engineer has for understanding the shear strength of soils is the effective stress principle. This principle is generally used by engineers to predict shear strength but it can also be used to provide an insight into the fundamental causes of shear strength of soils. The effective stress principle has been neglected until now in chemical stabilization work in favor of easier quicker methods which, although sometimes reliable, have not provided the tools so necessary for a fundamental analysis of what the stabilizers actually do to the soil. By understanding how stabilizers interact with soil to generate greater shear strength, one is, of course, in a better position to evaluate the effectiveness of a stabilizer and to develop more effective and more versatile stabilizers.

The unconfined compression test is often employed for evaluation of stabilizers. The strength measured by such a test could be caused by negative pore pressures set up during shear which increase the effective stress acting on the sample and hence the shearing resistance. If this same sample were sheared slowly in contact with water, the shearing resistance would be much lower since the negative pore pressures would be dissipated. On the other hand, if the unconfined strength of a stabilized soil were caused primarily by cementing of particles, i.e., by a "true cohesion," rather than by negative pore pressures, this second sample would retain its strength even if sheared slowly under water. The standard unconfined compression test obviously cannot differentiate between these fundamentally different shear mechanisms which may, in many cases, be of great practical concern. Shear tests in which pore pressures are measured and the effective stresses determined would, of course, show which of the above types of shear mechanisms predominated. This simple example illustrates how the effective stress principle can be used to obtain both practical and fundamental information.

A knowledge of the strength behavior of stabilized soils in terms of effective stresses does not, however, provide the complete picture. In order to ascertain what a stabilizer does, one should not only measure the end product but also the change in strength behavior caused by the stabilizing agent. In other words, the behavior of the "blank", i.e., an unstabilized compacted sample, in terms of effective stresses is required. Such knowledge will not only help to interpret the effects of stabilization, but will also assist in a better understanding of compacted soils in general. The practical advantages are obvious.

This section presents the results, which are of a preliminary nature, of the work thus far accomplished in this new area of research dealing with effective stresses in stabilized soils. The first two parts concern effective stresses and strength in a partially saturated unstabilized compacted cohesive soil. The third part presents data on the strength of a saturated stabilized clay in terms of effective stresses.

B. "Residual Pore Pressure Measurements in Soils"--M.S. Thesis in Civil Engineering by P. Bourque, C.S.C., 1961

The purpose of this thesis was to develop an accurate, reliable device for measuring negative pore pressures and to use this device for establishing relationships between air pressure, water pressure, and water content in a partially saturated soil.

The soil used for this investigation was a laboratory-prepared mixture of 80% silt (-200 mesh quartz called Potters flint) and 20% clay (a kaolinite called Peerless Clay). This mixture was chosen because preliminary tests indicated that it provided measurable pore water tensions over a large range of water contents.

The apparatus used for measuring negative pore pressures is shown in Figure VII-I. The sensing element is a fine ceramic disc (3.5 mm. thick x 30 mm. dia.) set and bonded into the base of a triaxial cell (Clockhouse cell for 1.4" dia. sample) with epoxy glue. The stone has an air entry value of 60 psi. The recording instrument is a pressure transducer. It is excited by a 6-volt battery and its output signal is transmitted to a sensitive millivoltmeter. An air cylinder is used for pressuring the triaxial cell. A means of applying water pressure to both the cell and the pore pressure lines is also provided.

A large number of instrumentation checks were made to ensure that the device would perform as expected. It was found that a careful de-airing technique was essential for removing entrapped air from the base pore pressure lines. All connections had to be absolutely airtight.

Compacted samples at various water contents were prepared by kneading compaction in a Harvard miniature mold. An effort approximating Proctor compaction was used in which three layers of soil were kneaded 25 times each with a 40-lb. spring tamper.

The residual pore water tension (u_r) was measured by sliding the compacted sample carefully onto the ceramic disc. If the tension approached one atmosphere, the cell chamber was clamped onto the

base and a lateral air pressure was applied to the sample to prevent cavitation. The output of the pressure transducer was read on the millivoltmeter and converted to pressure units.

Direct measurements of negative pore pressures were made up to -0.9 atm. Larger values of pore water tension (u_c) were measured indirectly by applying an all-around air pressure (u_a) to the sample large enough to make the water pressure (u_w) greater than -0.9 atm. The residual negative pore pressure was then considered to be the algebraic difference of the applied air pressure and the measured water pressure, i.e., $u_c = u_a - u_w$.

It was found, however, that an increment of applied air pressure results in an equal increase of water pressure only when the sample is dry of optimum. For this soil, the "response", measured as $\Delta u_w / \Delta u$ where Δu is an increment of air pressure, was 100% only when the samples were 3% or more dry of optimum. The response was lowest (91%) at optimum. One hundred per cent response is obtained only when most of the air in the sample is interconnected (or when the sample is 100% saturated). An increment of air pressure then causes no volume change and is, therefore, transmitted directly to the pore water. If much of the air is in bubbles, the applied pressure will compress these bubbles allowing the soil skeleton to consolidate under the increased stress ($u_a - u_w$). When this occurs a response of less than 100% is measured. Tests 7 and 8 (Table VII-1) illustrate this point. Sample 7 was compacted at optimum. The initial pore pressure response was only 91%. After applying the air pressure for twenty hours, the response had increased to only 94%. In test B the initial pore pressure response was 97% and did not increase during 11 hours of applied air pressure.

The results of the pore pressure measurements versus water content are plotted in Figure VII-2 along with the results of unconfined compression tests. Table VII-1 shows the effect of applied air pressures on the measured values of pore water pressure.

The availability of a simple reliable device for measuring residual pore water pressures up to -60 psi should prove very valuable in further research on partially saturated soils. Triaxial shear tests can be run using this device measuring pore pressure changes during shear.

This thesis demonstrates that caution must be used when applying an all-around air pressure to measure pore water tensions below -1 atm. Reliable results may not be obtained unless the sample is at least 2% dry of optimum.

C. "Residual Pore Pressures and Shear Strength of Compacted Clay"--
S.B. Thesis in Civil Engineering by S. K. Gulhati, 1961

Tests were run on three laboratory-prepared mixtures of silt (-200 mesh quartz) and clay (Peerless Clay). Correlations were obtained between molding water content, dry density, residual pore pressure, and undrained shear strength.

All samples were prepared by kneading compaction in a Harvard miniature mold using 3 layers with 25 blows per layer with a 40-lb. spring-loaded tamper.

Residual pore water pressures were measured using the device described in Part B. The relationship between molding water content and residual pore pressure for the three soils is shown in Figure VII-3.

One of the soils, a mixture of 40% kaolinite and 60% quartz, was used for a series of consolidated undrained triaxial tests. The samples were saturated by back-pressuring the pore water. The effective consolidation pressure ($\sigma_3 - u$) was made equal to the residual pore water tension measured after compaction. It was felt that this would provide a minimum of structure disturbance. Thus by compacting samples to the same density dry and wet of optimum moisture content, the influence of structure on effective stress shear parameters could be investigated. The stress vectors for the tests are shown in Figure VII-4. All tests reach the same shear envelope at failure, with a ϕ of 36.2° (Figure VII-5). No large difference was noted between tests dry and wet of optimum. This may have been due to disturbance of structure during the saturation process, particularly on the dry-side samples where compaction often results in a meta-stable structure which is very sensitive to changes in water content.

D. "Effective Stress Shear Parameters for a Clay Stabilized with Lime"--S.B. Thesis in Chemical Engineering by L. G. Bromwell, 1961.

This investigation determines the effect of hydrated lime (Ca(OH)_2) on the shear parameters of a fat clay (Vicksburg Buckshot Clay) in terms of effective stresses. Eight consolidated undrained (CU) triaxial tests with measured pore pressures were run on compacted samples of Vicksburg Buckshot Clay plus 5% reagent grade hydrated lime. Comparison tests were run on similarly prepared samples in unconfined compression.

Preliminary tests were run on Vicksburg Buckshot Clay plus 5% portland cement. The effect of consolidation pressure on strength

was quite small for this system at the pressures available with conventional triaxial equipment (150 psi) so that the results were inconclusive. These data are not reported.

Triaxial specimens were prepared in a Harvard miniature size mold by two-end static compaction in a hydraulic press. The compaction pressure used (about 57 kg./cm.²) was that necessary to give a dry density of 96 lb./ft.³. The molding water content was 21%. Optimum water content was 26%. All samples were cured for one week at 100% R.H. and room temperature. They were then immersed in distilled water for twenty-four hours. During soaking the degree of saturation increased from about 75% to 91%.

After soaking the samples were placed in a triaxial cell and allowed to consolidate for one day. Filter strips were used to facilitate drainage on six of the tests. They were not used in two of the tests. After consolidation, the samples were back-pressured for twenty-four hours to dissolve the remaining air. This usually required 4 kg./cm.² water pressure. Tests were then run with constant pore pressure, the chamber pressure being varied to maintain constant volume. A nominal strain rate of 1% per hour was used. The tests were performed with N.G.T. triaxial cells, pressure units, and load frames.

It should be noted that these tests were run on saturated samples. Normally, strength tests for stabilized soils are run on samples which are only partially saturated. For example, the unconfined tests (which were not back-pressured) were run at an average degree of saturation of 91%.

The triaxial test results are summarized in Table VII-2. Stress-strain data are plotted in Figure VII-6. The samples failed at low strains which is typical for "dry-side" samples. A comparison of the strength of Vicksburg Buckshot Clay plus 5% lime with that of the natural soil (Figure VII-7) shows the stabilized soil to be appreciably stronger. At the same void ratio and water content, the lime-stabilized soil is twice as strong as the natural Vicksburg Buckshot Clay. Part of this strength increase is probably due to prestressing or overconsolidation during the compaction process. The vector curves (Figures VII-7 and 8) are typical of overconsolidated samples having an overconsolidation ratio of about 6 to 12.

The major conclusions drawn from this investigation are the following:

- (1) Stabilization results in increased strength. At the same void ratio and water content, lime stabilized VBC is twice as strong as the unstabilized soil.
- (2) Lime stabilization of VBC results in a high cohesion intercept (1.6 kg. / cm.^2) and an increase in friction angle from 23° to 32.5° (Figures VII-7 and 9). These effects are thought to be caused by increased bonding between particles. Further testing may indicate to what extent this bonding is stress-independent (true cohesion) and to what extent it is stress-dependent (increased friction angle).
- (3) The mobilization of shear strength in stabilized soil does not require the development of large negative pore pressures. This was shown by test 2X-1 which was, in effect, an unconfined test with measured pore pressure. The pore pressure remained zero ($\pm 0.03 \text{ kg. / cm.}^2$) throughout the test (Table VII-2, Figure VII-8).
- (4) Stabilization increases the rigidity and reduces the compressibility of a plastic soil.

It is recommended that the generality of the results and conclusions derived from this investigation be checked by similar tests on different soil-stabilizer systems.

Tests should be run using higher pressures to provide better correlation with triaxial tests on natural clays and to permit testing of more rigid systems. Significant additions to and correlations with this thesis could also be obtained by running similar tests on partially saturated samples, measuring both water and air pore pressures. This would provide further insight into the relative importance of various components of shear strength and compressibility. Such an approach is also more realistic, since it studies stabilized soils as they are generally employed--in a partially saturated state.

TABLE VII-1
Residual Pore Water Tension in Compacted Soil

Water Content (%)	Test No.	u _a (1)	Pore Water Tension, u _c (atm)						Av. Response u _c (atm)	Average Corr.	(3)	
			0	1	2	3	4	3	2	1	0	
1	12.8	.40	1.09	1.09	1.05	1.08	1.10	1.08	1.09	1.09	100%	1.08±.03
2	14.9	.53	.88	.92	.93	.95	.94	.99	.98	.88	100	.96±.03
3a	16.5	.59	.63	.68	.66	.68	.67	.68	.63	100	.67±.01	.94
3b (5)	16.5	.59	.67	.68	.67	.68				100	.67±.01	1.00
4	17.0	.64	.58	.61	.61	.63	.62	.63	.60	100	.61±.02	.95
5	17.4	.69	.36	.46	.48	.52	.51	.50	.47	.46	.38	99
6	18.5	.80	.30	.34	.35	.40	.40	.37	.36	.36	.35	99
7a	20.0	.90	.04	.14	.23	.32	.32	.19	.05	91	.22±.10	.23
7b (5)	20.0	.90	.07			.30				94		
8a	21.0	.92	.05	.10	.12	.15	.19	.14	.08	.06	97	.13±.06
8b (5)	21.0	.92	.06	.15	.15	.20	.24	.19	.13	.10	96	.16±.08
9	25.9	.96	.01	.03	.06	.08	.11	.06	.03	.01	.01	.06±.05

(1) Triaxial cell air pressure (atm)

(2) The average of results obtained by the indirect method of measurement (atm), i.e., when $u > 0$ atm

(3) Correlation between direct and indirect methods of measurement = u_c at $u_a = 0/u_c$ at $u_a > 0$

(4) Response of the residual pore water pressure to an increment of ce
 (5) Samples 3, 7, and 8 tested 17, 20, and 11 hours later respectively

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TABLE VII-2
 Summary of Test Data
 Note: All stresses in kg. / cm.²

Test No.	As Molded			Final			at $(\sigma_1 - \sigma_3)$ max.							
	w (%)	S (%)	γ_d (pcf)	w (%)	S (%)	e	$\bar{\sigma}_c$	Pore Pres.	σ_f (%)	$(\sigma_1 - \sigma_3)$	$\bar{\sigma}_1 / \bar{\sigma}_3$	$\Delta u / \sigma_1 - \sigma_3$	$\frac{\sigma_1 - \sigma_2}{2} \frac{\sigma_1 + \sigma_3}{2}$	
2X-1	21.2	76.5	96.1	27.9	100	0.74	0	94%	0.32	6.20	156	-.006	3.10	3.14
2X-2	21.0	74.6	95.4	27.45	97.7	0.76	1.00	>80%	0.71	8.49	8.9	-.005	4.24	5.32
4X-1	20.6	74.7	96.0	27.8	100	0.74	1.00	88%	1.07	7.96	11.2	.028	3.98	4.76
1X-1	20.9	75.5	96.0	27.4	98.7	0.74	2.00	80%	0.68	9.71	7.47	.155	4.85	6.36
1X-2	20.75	76.2	96.5	26.8	98.4	0.73	4.00	80%	0.63	9.96	6.15	.476	4.98	6.90
3X-1	19.1	71.4	97.5	26.1	97.5	0.72	6.00	77%	0.64	11.62	5.93	.314	5.81	8.17

AYOUT FOR THE MEASUREMENT OF NEGATIVE PORE-WATER PRESSURE

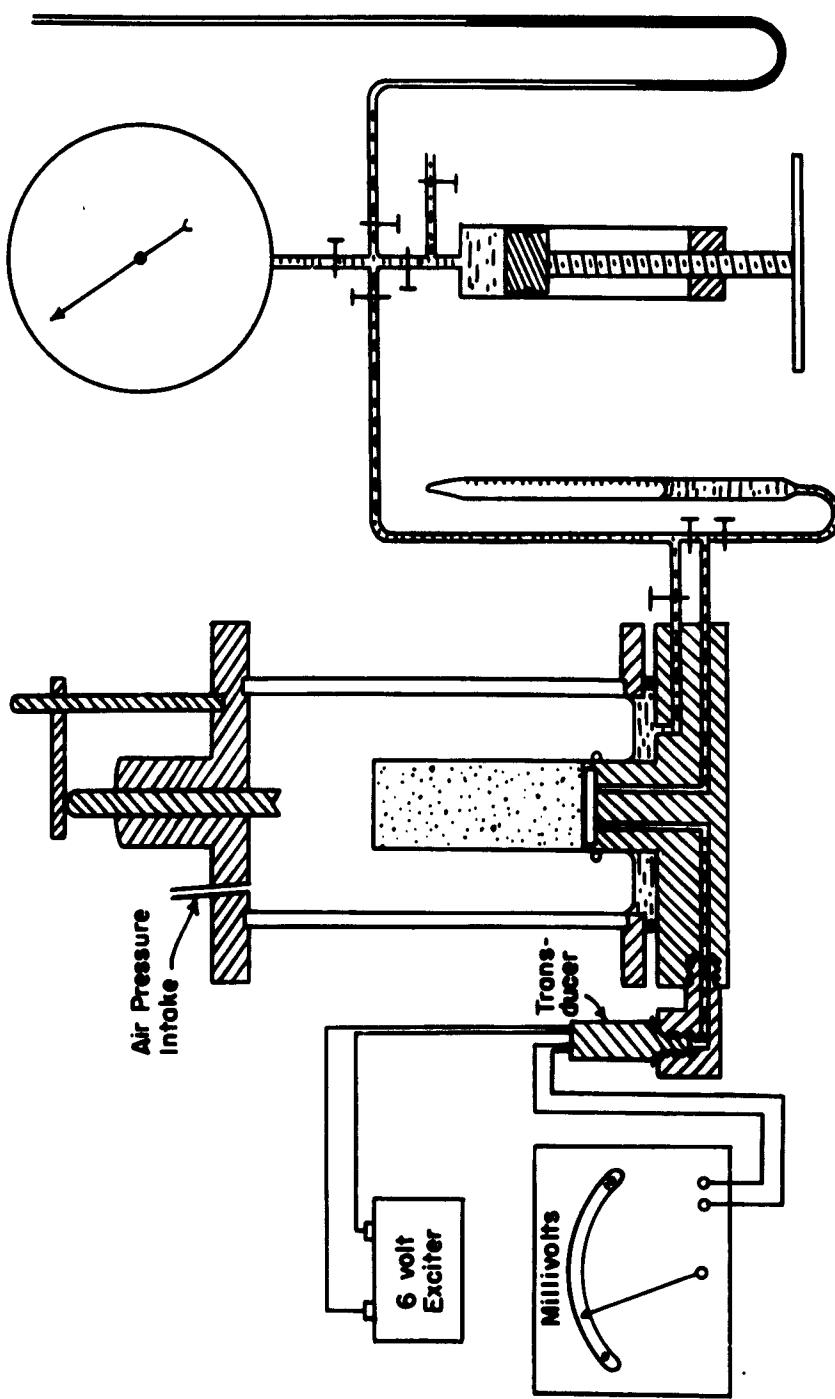


Figure VII-1

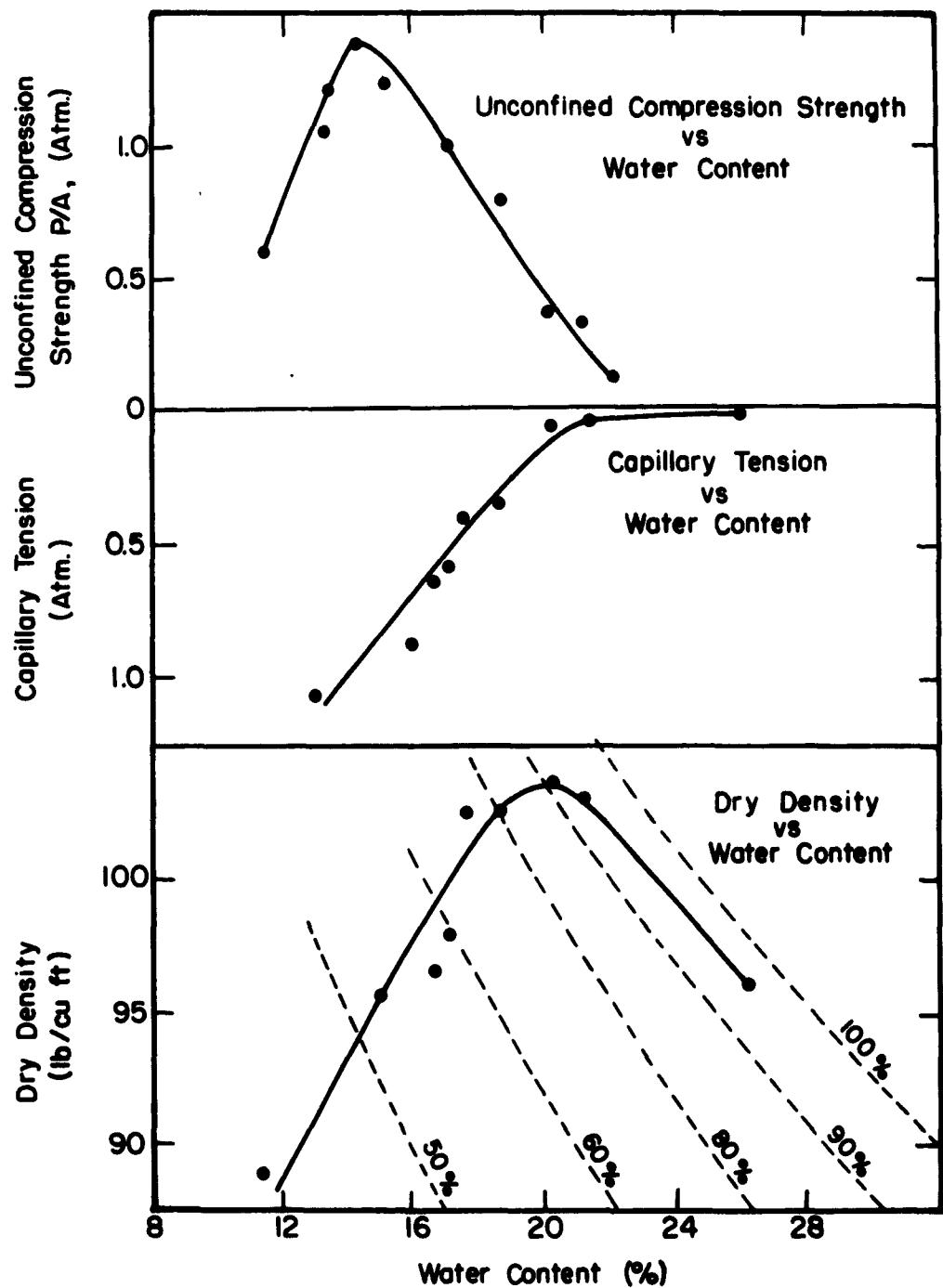


Figure VII-2

DRY DENSITY AND RESIDUAL PORE PRESSURE TENSIONS
vs MOLDED WATER CONTENT

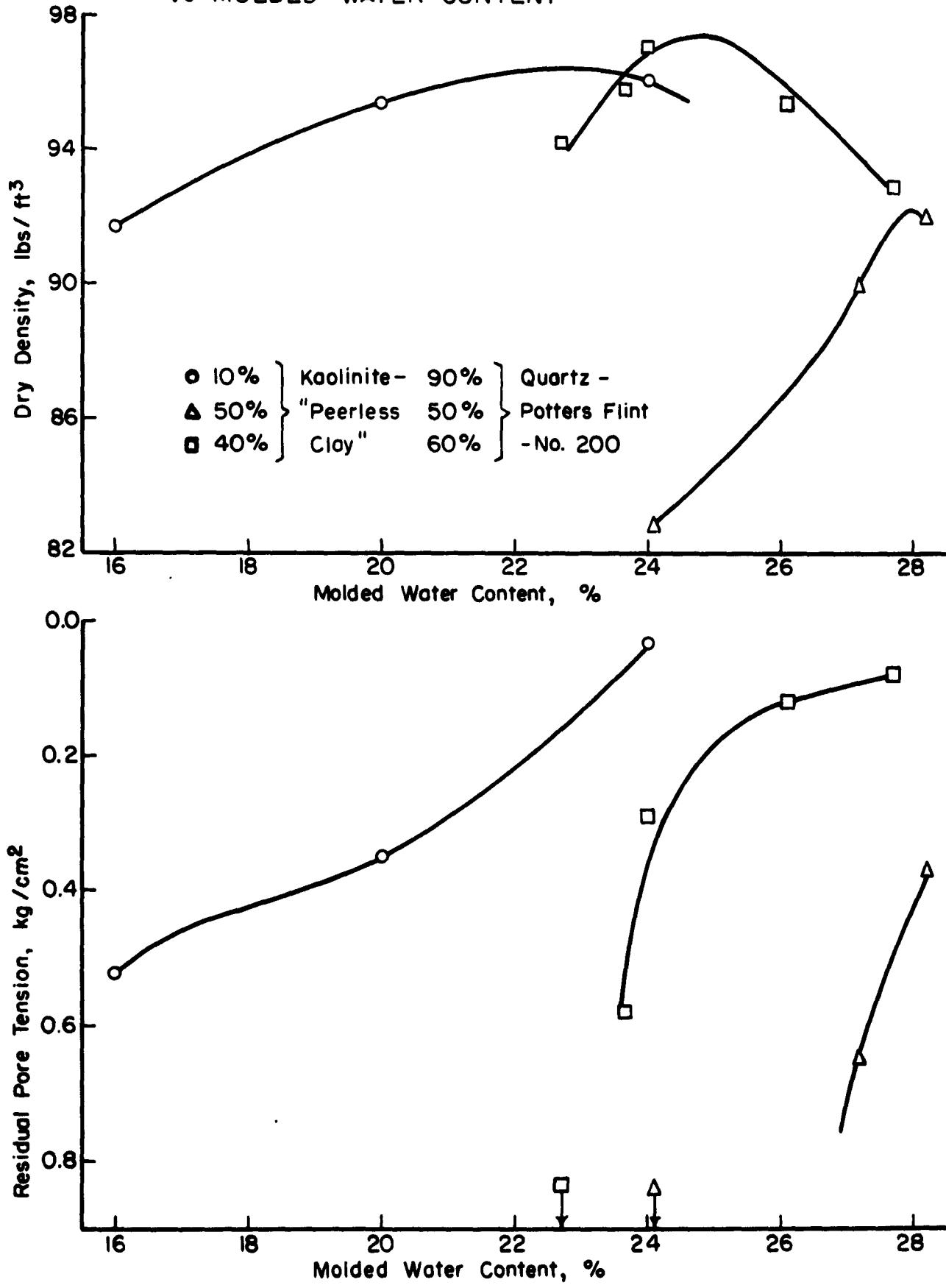


Figure VII-3

VECTOR CURVES - CU TRIAXIAL TESTS

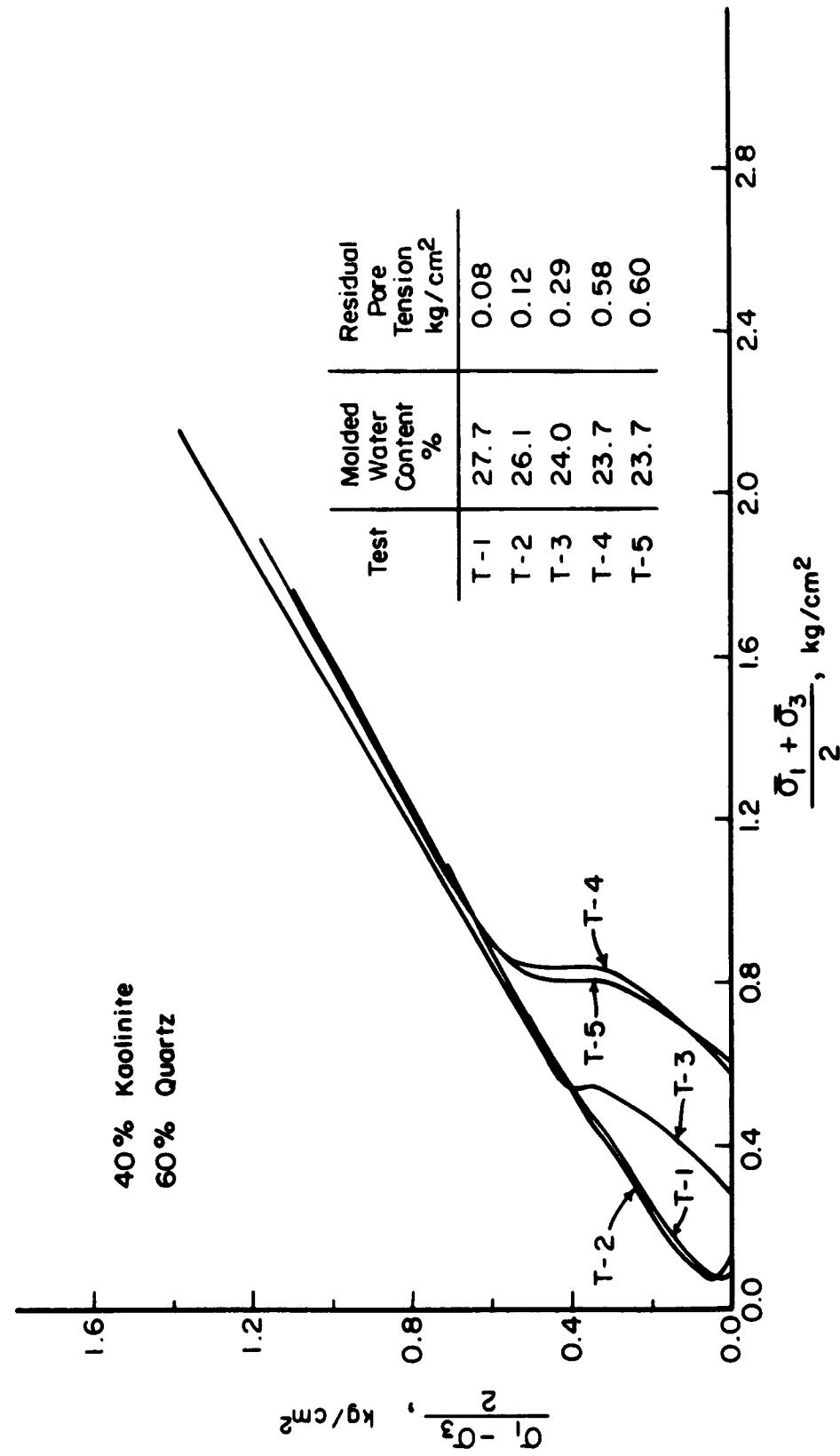


Figure VT-4

EFFECTIVE STRESS ENVELOPE - CU TRIAXIAL TESTS

(For stresses at 10% axial strain)

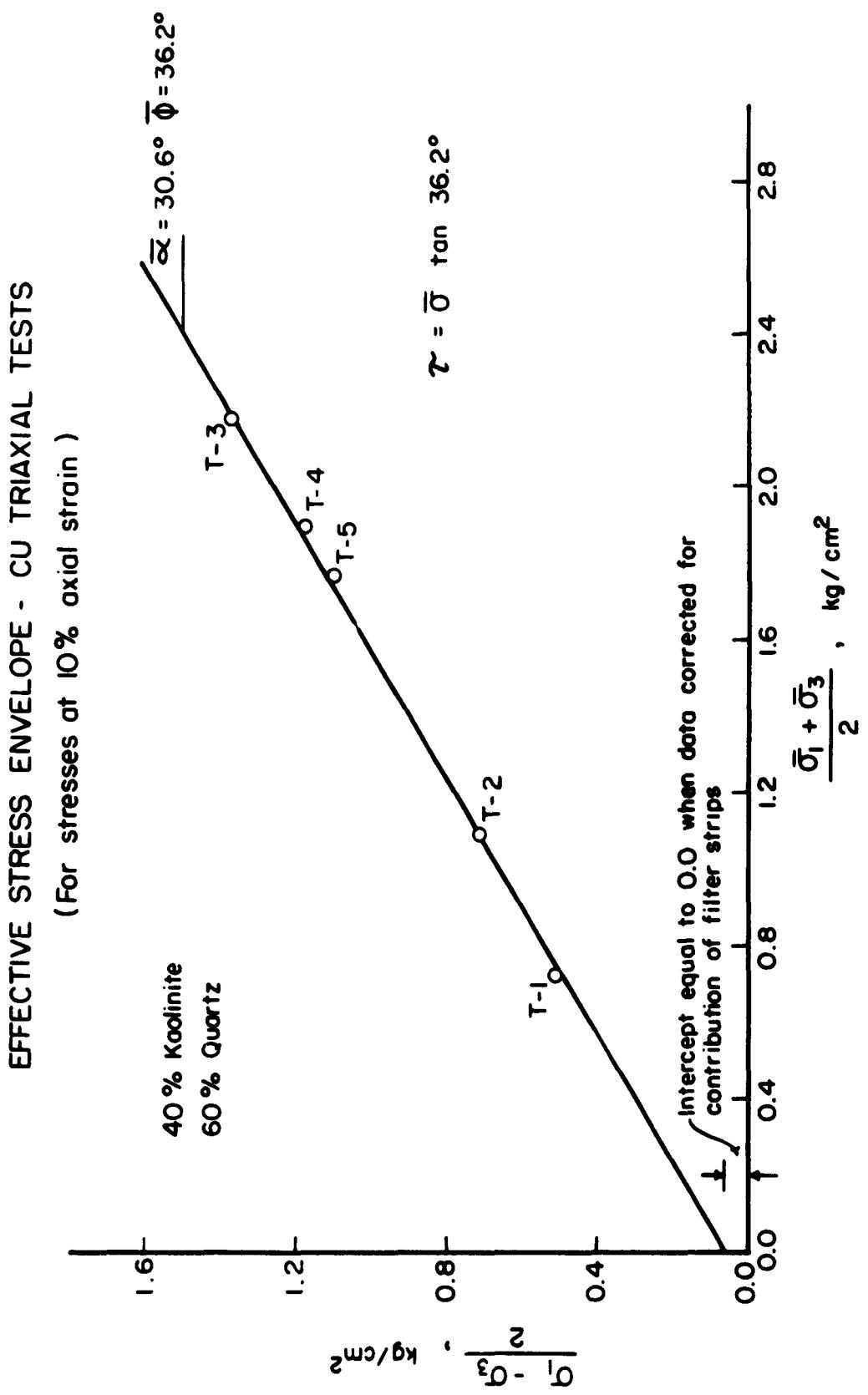


Figure VII-5

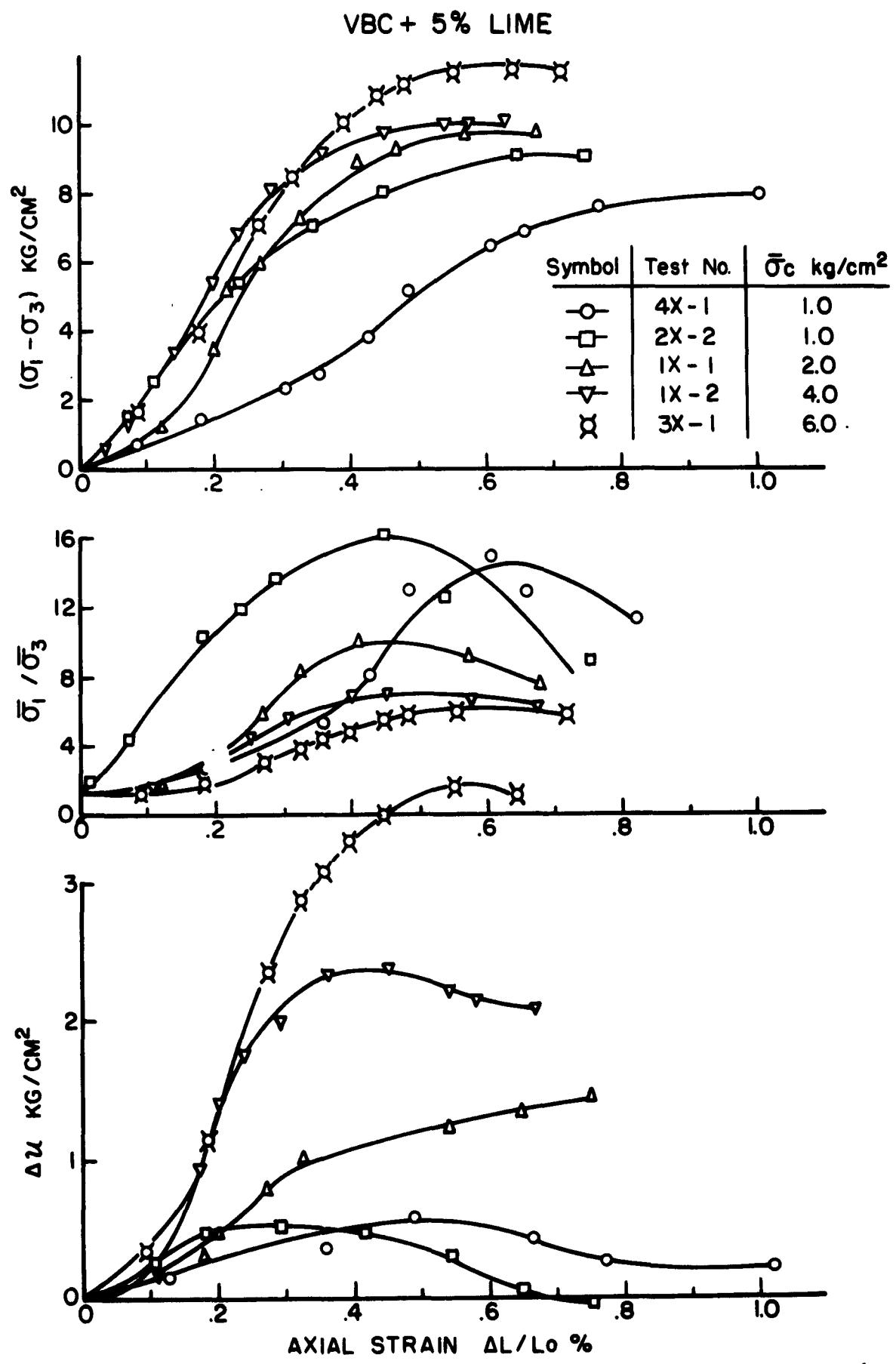


Figure VII-6

COMPARISON OF VECTOR CURVES FOR NATURAL
AND STABILIZED VBC
45° LINES ARE LINES OF ZERO PORE PRESSURE

Symbol	Types of Soil	Maximum Post Pressure	$\bar{\sigma}_c$ kg/cm ²
■	Stabilized	57 kg/cm ²	1.0
▼	Stabilized	57	4.0
✖	Stabilized	57	6.0
●	Natural	6.19	0.5
○	Natural	6.19	1.0
○	Natural	6.19	6.19

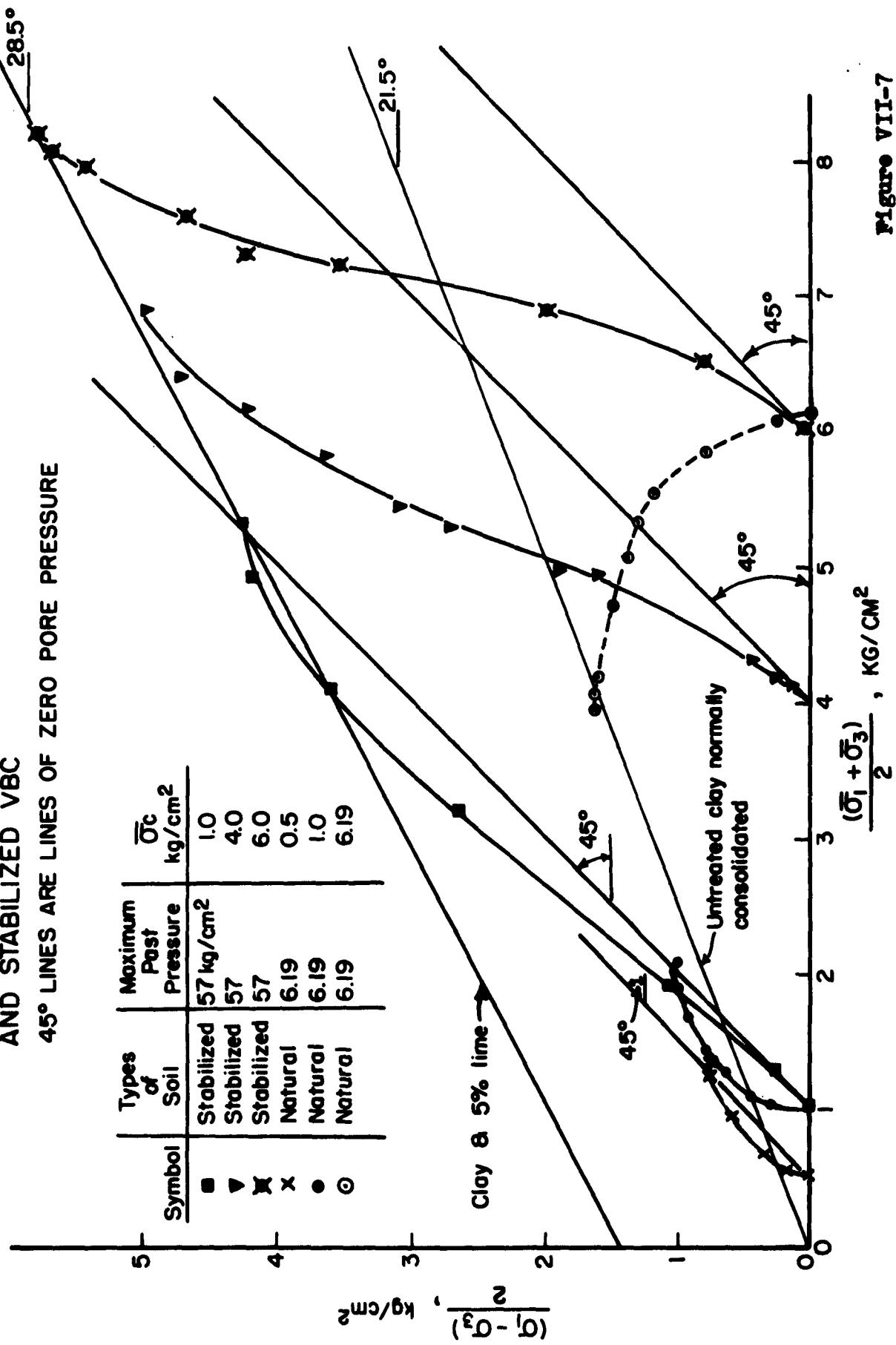


Figure VII-7

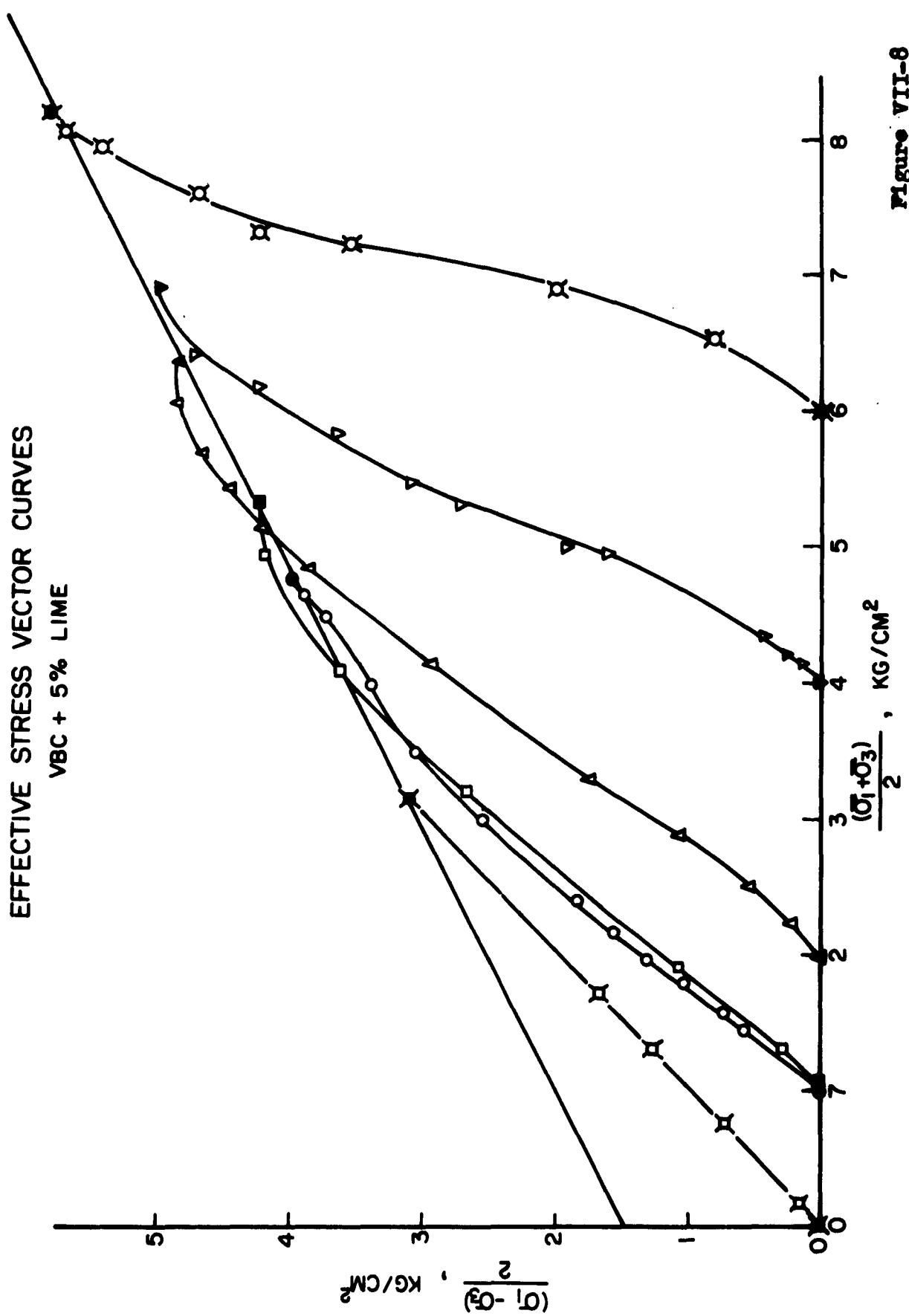


Figure VII-8

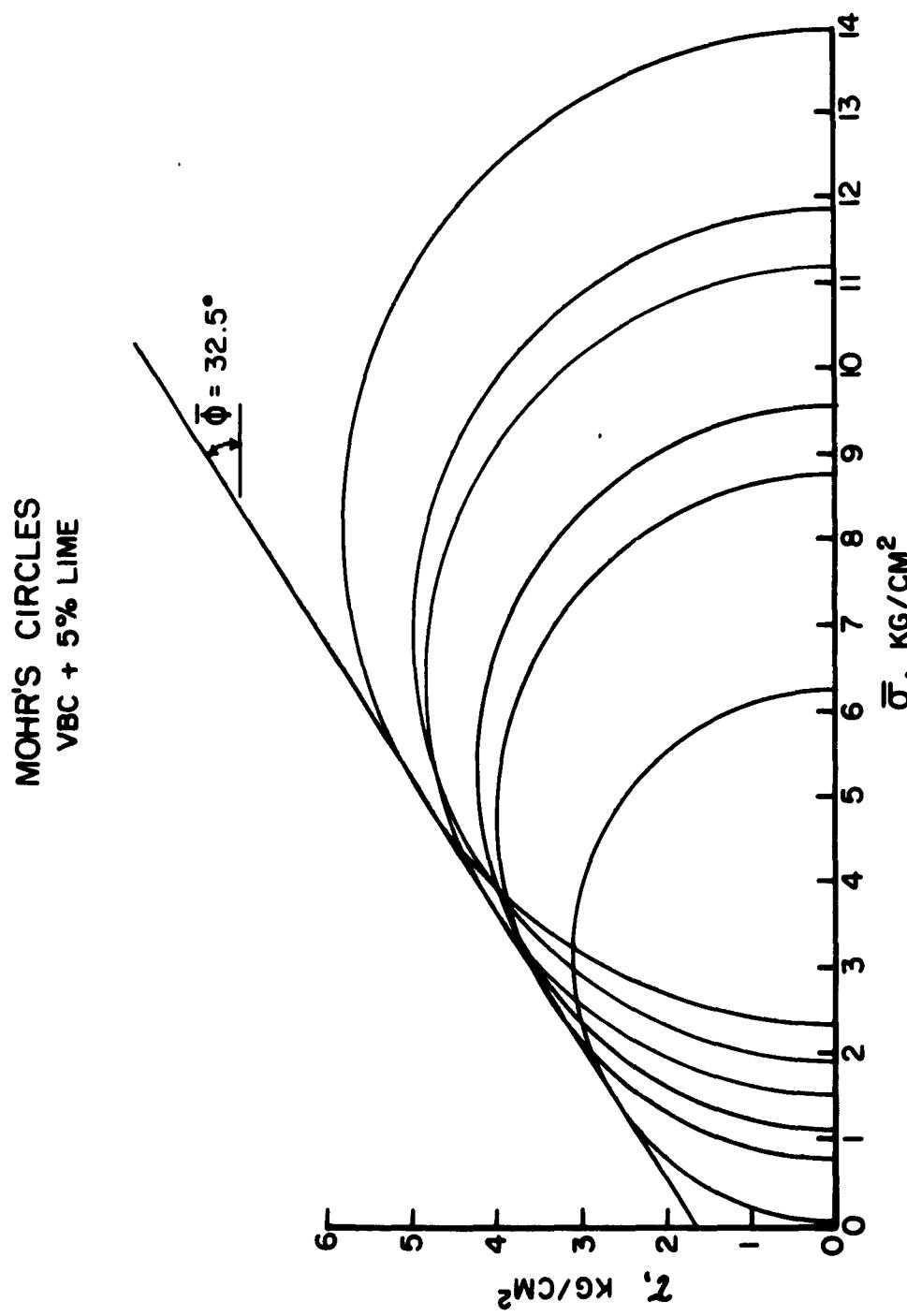


Figure VII-9

VII. THESES COMPLETED IN 1961

The following are abstracts of theses completed at M.I.T. in 1961 which deal with subjects of interest in soil stabilization.

A. Permeability and Shear Strength of Kaolin with Ethylene Glycol and Formamide as Pore Fluids--S. M. Thesis in Dept. of Civil Engineering by Raymond F. Friescke, 1961

The object of this thesis was to observe some of the changes in the engineering behavior of kaolinite under the influence of different pore fluid viscosities and dielectric constants.

To accomplish the above, the following data were obtained: liquid limits, shrinkage limits, kneading and static compaction curves, unconfined compressive strength, permeabilities, and undrained triaxial shear strength with pore pressure measurements.

With the exception of the permeability and triaxial tests, the samples were mixed or molded with the different pore fluids. In the permeability tests, four samples were compacted with water and water was allowed to permeate through them until the flow reached a constant rate. The permeation fluids of two of the samples were then changed to ethylene glycol and formamide. It was believed that when the different pore fluids were introduced in this manner, the fabric of the samples would remain relatively unchanged. The reproducibility of the water samples was excellent. In those samples in which the permeant was changed, the permeability changes were not proportional to the change in permeant viscosity. The permeability of the compacted kaolinite to glycol was only 16.7 times less than that for water, whereas the viscosity was larger by a factor of 19.5. The permeability with formamide was 10.6 times less than that for water whereas it should have been only 3.8 times less. The results obtained with glycol were taken as an indication that there are no highly viscous fluid layers on the surface of kaolinite. The results obtained with formamide were believed to be due to the adsorption of formate anions on the edges of the clay which had a dispersing effect on enough of the particles to lower the permeability.

The four samples used for the permeability determinations were extruded and subjected to an undrained triaxial shear test with pore pressure measurements. Since the initial fabrics were predominantly the same, any differences in strength behavior should have been due to the properties of the pore fluids. As can be seen in Figure 10, the glycol sample was about three times stronger than the water samples and the formamide sample was about 1.5 times stronger.

The glycol sample showed a large negative* pore pressure at the end of the test; the pore pressure of the formamide sample was somewhat less negative. In comparison, the pore pressure of the water samples was slightly positive at the end of the test. At the same time, all samples yield about the same value of ultimate obliquity of stresses.

A detailed explanation of the strength data has not yet been formulated although it seems fairly obvious that the viscosity of the pore fluid must play an important role in the development of pore pressures and strength. Work in this general area of research is continuing.

B. Thixotropic Behavior of Laurentian Clay--S. B. Thesis in Civil Engineering by W. T. Jackson, Jr., 1961

A large batch of Laurentian clay (illitic clay; liquid limit = 56%, plasticity index = 30.5%; 70% clay size) was consolidated one-dimensionally to 0.75 kg/cm^2 from a slurry (300% water content, very low pore water electrolyte concentration). The batch was then extruded and samples immersed in transformer oil (to maintain a constant water content) for various times (one half to 24 days) before testing by stress controlled unconsolidated-undrained triaxial tests with pore pressure measurements.

The principal findings were that:

- (1) The strength (deviator stress at 0.6% axial strain) increased from 0.25 kg/cm^2 to 0.40 kg/cm^2 after 24 days of aging at constant water content.
- (2) The residual pore pressure before shear decreased from -0.16 kg/cm^2 to -0.30 kg/cm^2 at the end of 24 days.
- (3) The increase in strength with time was uniquely correlated with the decrease in residual pore pressure (i.e., increase in effective stress) with time.
- (4) All samples had approximately the same values of $(\sigma_1 - \sigma_3)/\bar{\sigma}_o$, $\Delta u/\bar{\sigma}_o$, and $\bar{\sigma}_1/\bar{\sigma}_3$ ratios versus axial strain during shear wherein:

* The triaxial tests were actually constant volume tests in which the chamber pressure was varied to maintain constant volume.

$(\sigma_1 - \sigma_3)$ = deviator stress

σ_o = initial effective stress = minus residual pore pressure

Δu = change in pore pressure during shear

σ_1 = major principal effective stress

σ_3 = minor principal effective stress

(5) The shrinkage limit of the samples after shearing did not change with time.

The results might be explained by a build-up of "water-structure" around the particles with time that decreased the free energy of the water in the soil and hence caused the decrease in pore pressure. A somewhat less likely explanation is that the clay particles wanted to reorient themselves into a more flocculated fabric with time. The data show that cementation (due to amorphous silica, alumina, etc.) probably did not contribute to the strength increase with time.

C. Effects of Salt Concentration on the Shear Strength of Boston Blue Clay--S. B. Thesis in Civil Engineering by William A. Bailey, 1961

Consolidated-undrained triaxial tests with pore pressure measurements were run on three types of Boston Blue clay: Samples consolidated from a slurry prepared with water (water samples); samples consolidated from a slurry prepared with a NaCl concentration of 35 g/l (salt samples); and salt samples leached (actually diffused) with water for various periods of time after consolidation to 3.0 kg/cm² (leached samples).

The data showed:

- (1) Water samples had a lower consolidation curve (by about 2% water content) than salt samples but the same S_u / σ_c ratio (S_u = undrained strength, σ_c = consolidation pressure).
- (2) Water samples had a slightly lower (28.5 versus 30 degrees) effective stress envelope than salt samples for normally consolidated samples.
- (3) Leaching the salt out of salt samples:
 - a) Lowered S_u and the strain at failure and increased the sensitivity.

- b) Increased Skempton's A factor and Δu (pore pressure) and lowered $(\sigma_1 - \sigma_3)$ (deviator stress) and $(\bar{\sigma}_1 / \bar{\sigma}_3)$ (principal stress ratio) for equal values of axial strain. Figure VIII-2 shows a typical plot of the effects of leaching on the stress vs. strain behavior of Boston Blue clay.
- c) Lowered the effective stress envelope for $(\sigma_1 - \sigma_3)$ max. from about 26 degrees to 13 to 21 degrees.

The data show that the lower strength, particularly at the larger strains, of the leached samples is due primarily to the very large excess pore pressure that was set up during shear. It is believed that an increased amount of particle reorientation caused the larger pore pressures in the leached samples. The increased reorientation in turn could be explained by the relative inability of particles to remake "contacts" during shear because of the increased double-layer repulsion that exists in the samples with the lower salt concentration.

D. A Study of the Effects of Environmental Changes on the Stress-Strain Properties of Kaolinite--M.S. Thesis in Civil Engineering
by Anwar E. Z. Wissa, 1961

Consolidated-undrained triaxial tests were run on three types of kaolinite (minus 2 microns) samples: samples sedimented and consolidated in water and then sheared (standard samples); as above but samples consolidated for several months at the same pressure prior to shear (aged samples); and aged samples which had 1 N NaCl diffused into the pore fluid prior to shear (salt samples).

The stress-strain data showed that*:

- (1) Aging had no effect on $\Delta u / \bar{\sigma}_c$ vs. strain but increased $(\sigma_1 - \sigma_3) / \bar{\sigma}_c$ at the lower values of axial strain, i.e., $(\sigma_1 - \sigma_3)_{\text{max.}}$ increased.

* Δu = change in pore water pressure
 $\bar{\sigma}_c$ = preshear consolidation pressure
 $\sigma_1 - \sigma_3$ = deviator stress (stress difference)
 ϕ = slope of the Mohr envelope which is tangent to Mohr circles in terms of effective stresses
 $\bar{\sigma}_1 / \bar{\sigma}_3$ = ratio of principal effective stresses

- b) Increased Skempton's A factor and Δu (pore pressure) and lowered $(\sigma_1 - \sigma_3)$ (deviator stress) and $(\bar{\sigma}_1 / \bar{\sigma}_3)$ (principal stress ratio) for equal values of axial strain. Figure VIII-2 shows a typical plot of the effects of leaching on the stress vs. strain behavior of Boston Blue clay.
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- (1) Aging had no effect on $\Delta u / \bar{\sigma}_c$ vs. strain but increased $(\sigma_1 - \sigma_3) / \bar{\sigma}_c$ at the lower values of axial strain, i.e., $(\sigma_1 - \sigma_3)_{\text{max.}}$ increased.

* Δu = change in pore water pressure
 $\bar{\sigma}_c$ = preshear consolidation pressure
 $\sigma_1 - \sigma_3$ = deviator stress (stress difference)
 ϕ = slope of the Mohr envelope which is tangent to Mohr circles in terms of effective stresses
 $\bar{\sigma}_1 / \bar{\sigma}_3$ = ratio of principal effective stresses

(2) The salt samples (compared to the aged samples) had a higher $\Delta u / \bar{\sigma}_c$ ratio during shear, a slightly lower value of $(\sigma_1 - \sigma_3)_{max} / \bar{\sigma}_c$ and a considerably lower $(\sigma_1 - \sigma_3) / \bar{\sigma}_c$ ratio at larger values of axial strain (i.e., the samples acted as if they were "sensitive").

The slopes of the effective stress envelopes were as follows:

	<u>System</u>	<u>$\bar{\phi}$, degrees</u>
At $(\sigma_1 - \sigma_3)_{max}$	Standard	24.5
	Aged	19.5
	Salt	17
At $(\bar{\sigma}_1 / \bar{\sigma}_3)_{max}$	Standard	27
	Aged	27
	Salt	24

All samples fell approximately on the same strength vs. water content line.

In addition to the above, the following data were also obtained:

- (1) Hvorslev parameters for standard samples
- (2) Effect of NaCl and NaOH concentration on sediment densities and Atterberg limits
- (3) Effects of time on the amount of extractable surface alumina in the kaolinite

E. Physico-Chemical Analysis of the Shear Strength of Saturated Clays --Sc. D. thesis in Civil Engineering by Charles C. Ladd, 1961

The thesis contains three principal parts. The first is a theoretical analysis of the physico-chemical forces acting in clay-water systems and a review of the importance of clay structure (the orientation and arrangement of particles, i.e., fabric and the interparticle forces) in determining the engineering behavior of clays. The second part reviews the shear strength behavior of saturated clays and the mechanisms that have been suggested to explain the fundamental cause of strength in both sands and clays. This is followed by a new hypothesis concerning the nature of strength generation in clays and reasons for their volume change and pore pressure behavior during

shear. The third part presents experimental data on the effects of time of consolidation, temperature, and pore fluid salt concentration, i. e., environment, on the strength behavior of saturated samples of a plastic montmorillonitic natural clay as measured by consolidated-undrained triaxial tests with pore pressure measurements. It also discusses some effects of environmental changes on the strength behavior of clays with different mineralogical compositions.

The following conclusions, as yet highly speculative, were drawn relative to the nature of interparticle forces, structure, and strength in clay-water systems. In the following, the terms "contact" and "double-layer" stresses are arbitrarily defined as stresses acting between particle surfaces separated by less than and greater than 20 Å respectively. "Mineral" contacts refer to actual mineral-to-mineral contacts wherein there is no water between the interacting surfaces and is one form of "contact" stress.

In most all normally consolidated clays (well dispersed sodium montmorillonite being an exception), most of the effective stress is transmitted via "contact" stresses and only a small fraction is carried by "double-layer" stresses. The "contact" stresses can exceed thousands of atmospheres. In turn, a large portion of the "contact" stress is probably transmitted through "mineral" contacts formed between the corner or edge of one particle and the face of another particle. The major role of "double-layer" stresses lies in their possible influence on clay fabric, particularly in dilute suspension and during shear. "Double-layer" stresses can be greatly altered by environmental changes.

Strength generation in clays can be broken into components of "true friction" and "true cohesion" as follows. The frictional component is caused by primary valence bonding at areas of "mineral" contact and is of the nature proposed by the Terzaghi-Bowden-Tabor theory of friction (as, for example, is believed to occur in sands, and between metal surfaces). The amount of "mineral" contact, and hence frictional resistance, will be a function of the effective stresses acting on the clay but may also be influenced by "double-layer" stresses and other types of "contact" stresses. The cohesive component of strength is caused by a net breakage of those interparticle contacts for which interparticle attractive stresses (van der Waals, electrostatic, etc.) exceed interparticle repulsive stresses (surface hydration, etc.) and/ or by the existence of cementitious bonds caused by carbonates, iron oxides, etc. The "structure" of water is not felt to be an important source of strength generation in clays, although it, like the "double-layer" stress, may influence the factors which do actually generate strength.

Pore pressures and volume changes during shear are believed to be controlled to a large degree by the net change in fabric (particle reorientation to a more dispersed fabric) during shear. The net change in fabric will depend on the initial fabric and on the ease with which "contact" stresses are reformed during shear. These factors are in turn influenced by the nature of the "contact" and "double-layer" stresses.

Experimental data on the effects of environmental changes on the volume, pore pressure and strength behavior of Vicksburg Buckshot Clay (P.I. * 40%) showed the following trends:

1. An increase in the temperature of a consolidated sample causes a relatively small volume decrease if the effective stress is maintained constant or a relatively large decrease in effective stress if the volume is maintained constant. The opposite occurs for a temperature decrease. A change in the magnitude of the "contact" stress rather than of the "double-layer" stress is believed to be responsible for this behavior.
2. The temperature changes prior to undrained shear had a relatively minor effect on the undrained strength and on the friction angle in terms of effective stresses. However, temperature changes during undrained shear could cause very large changes in the effective stress path.
3. An increase in the salt (CaCl_2) concentration of the pore fluid (either during sedimentation or by diffusion after consolidation) caused a relatively small increase in undrained strength and in the friction angle. The opposite was true if the salt concentration was reduced.

However, the effect of salt concentration on strength varies considerably with type of clay. For example, some univalent illitic clays (e.g., Norwegian quick clays) show very large strength decreases when the salt concentration is reduced whereas clay size univalent kaolinite may show a decrease in strength when the salt concentration is increased.

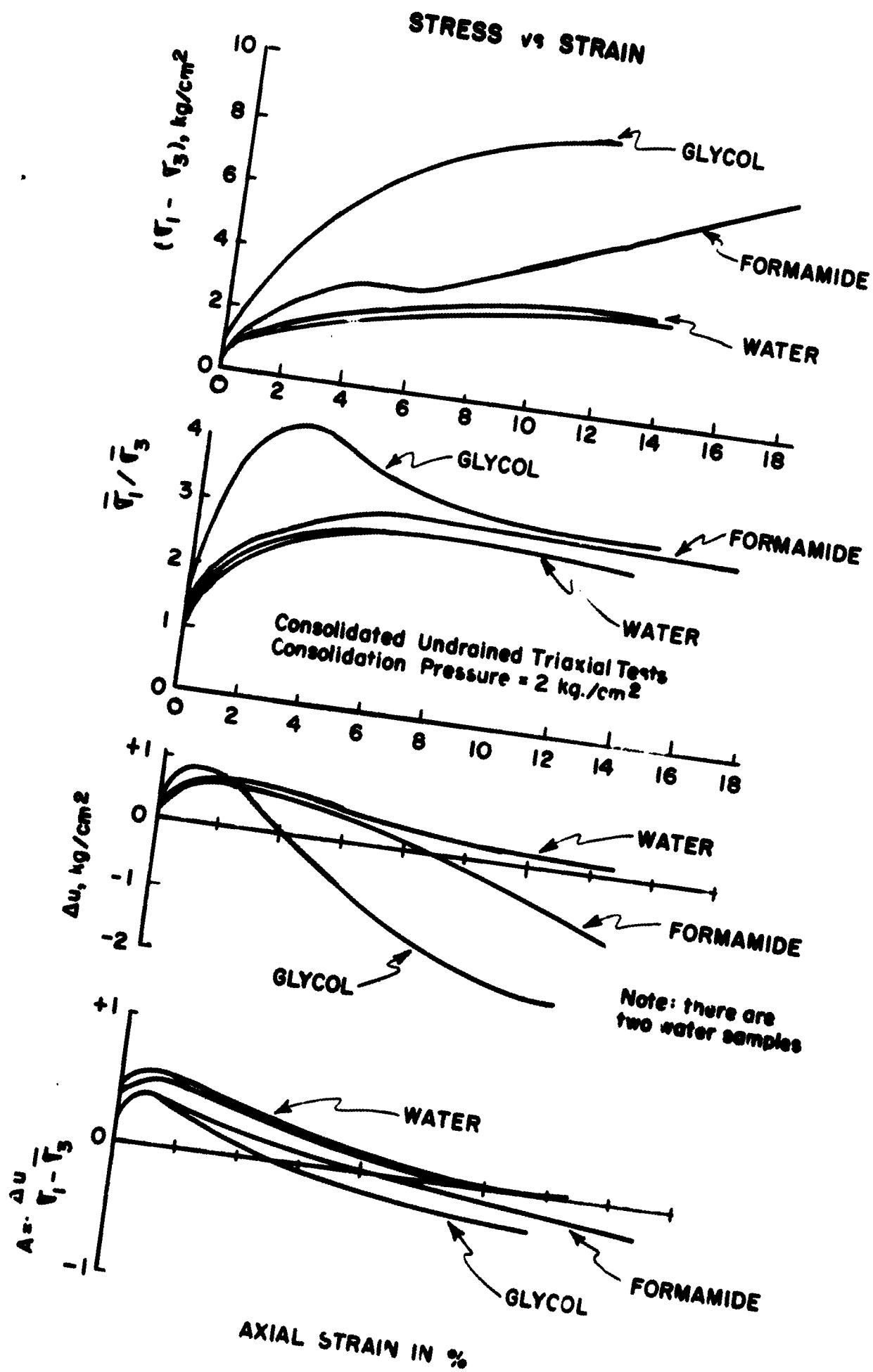


Figure VIII-1

EFFECT OF LEACHING SALT OUT OF BOSTON BLUE CLAY

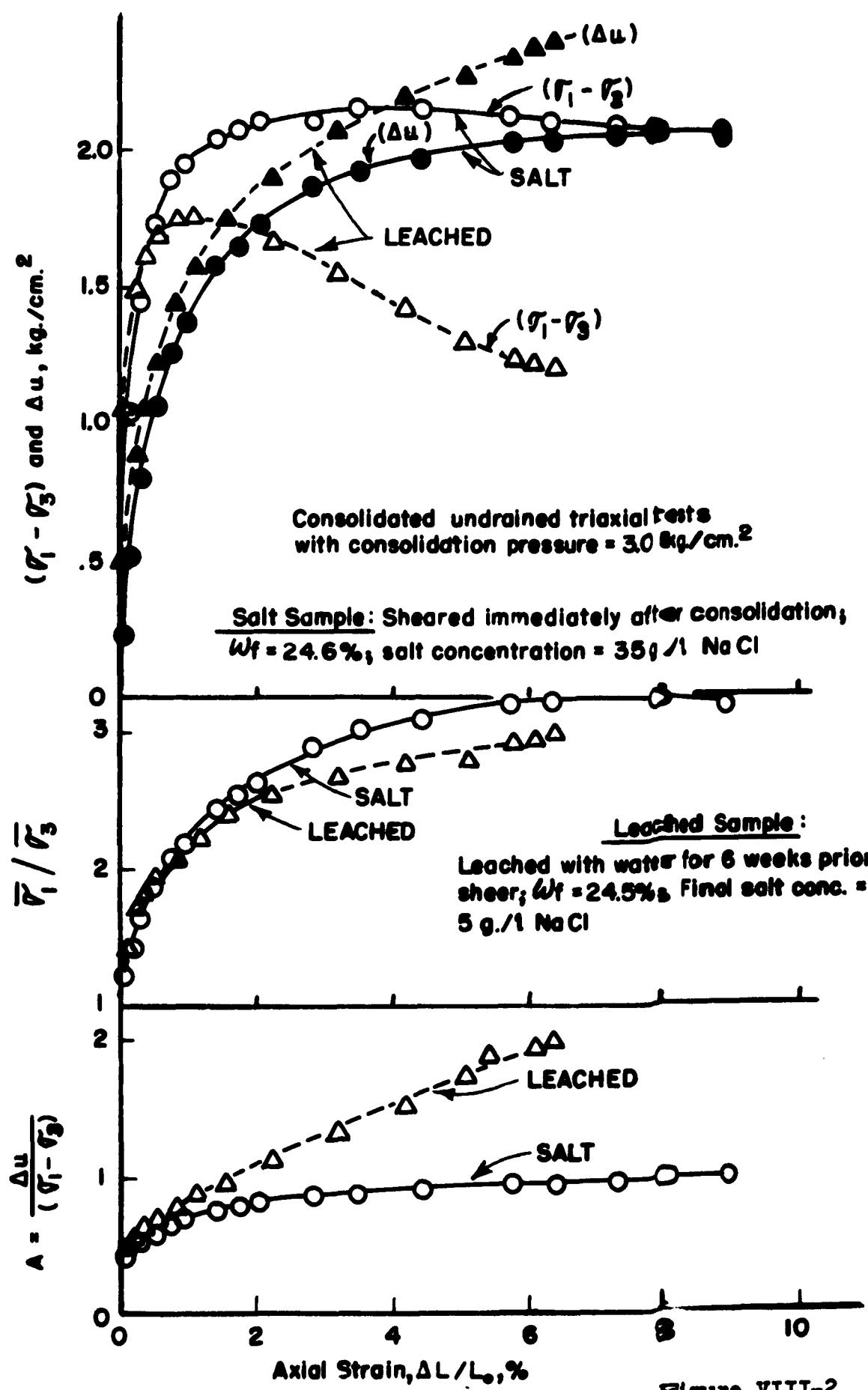


Figure VIII-2